

Chapter 2

Experimental Techniques

In this chapter we report on the experimental techniques used during this thesis both in the sample patterning and characterization. An important part will be focused on the lithography techniques and processes (micro and nano) used for the creation of structures with a well defined geometry in these scales. The setup of an experimental installation for the study of magnetotransport properties as a function of temperature, as well as that for electrical measurements inside a Dual Beam chamber, will be also explained. Spectroscopy (EDX and XPS), magnetometry (MOKE) and microscopy (SEM and AFM) techniques used during the work are included in this chapter. Most of the instruments described are located at the Institute of Nanoscience of Aragón (INA) and at the Institute of Science Materials of Aragón (ICMA). Spatially resolved MOKE and AFM were done over a two-month stage with the research group of Prof. Cowburn at Imperial College, London. Magnetotransport experiments at high static fields were performed at the High Field Magnet Laboratory of the University of Nijmegen, The Netherlands.

2.1 Lithography Techniques

Lithography is the group of techniques used to transfer previously determined patterns on a substrate. The typical size of these structures is micro- and nanometric. Depending on the type of lithography technique used, the transfer is performed in a different way, which defines the resolution that can be attained. In this thesis, we have used two large lithography groups, both of them within the top-down approximation:

1. Optical lithography: these techniques have been used in a clean room environment. This is by far the most common lithography technique in micro-electronic fabrication. It permits the fabrication of micrometric structures.

2. Lithography using focused electron and ion beams (Dual Beam system): with these techniques, nanostructures below 100 nm can be patterned.

We should remark that the equipment used was installed in the premises of the Institute of Nanoscience of Aragón (INA) in Zaragoza, during this thesis. Thus, the procedures for the majority of the processes that will be shown here were carried out by myself, together with the technical staff of the Lithography Laboratory (R. Valero, I. Rivas, and R. Córdoba), and Doctor Javier Sesé.

2.1.1 Optical Lithography

All the photolithography processes have been performed in a 10,000 class-clean room, at the INA. Clean rooms are special rooms with well-controlled ambient conditions. Temperature, humidity, differential pressure and flux of air, as well as lightning and electrostatic protection have to fulfill determined standards. Class = 10,000 means that the number of particles of size $\leq 0.5 \mu\text{m}$ per cubic meter should not be higher than 10^4 . A deviation of these parameters can alter the processes, as well as modify their quality.

We will give a short overview of the equipment and processes used by explaining one particular example patterned in this thesis: lithography in Fe_3O_4 thin films, whose results will be presented in [Chap. 3](#).

The starting point is to create a computer layout for the specific application. We have used the program Clewin[®] for this. In [Fig. 2.1](#), we show the mask design, where the different colors symbolize different layers. Since we wish to have two materials on top of the substrate (in this case MgO) with different shapes, it will be necessary to perform two lithographic processes. For both patterns to be well oriented one with respect to the other, lithography marks are added to the design (see [Fig. 2.1c](#)).

Once the pattern is done, two photolithography masks (one for each step) are made. These masks consist of a glass plate having the desired pattern in the form of a thin ($\sim 100 \text{ nm}$) chromium layer. The masks were manufactured by Delta Mask[®], company located in the Netherlands.

As shown in [Fig. 2.2](#), the lithography process involves the following steps:

First step: The Fe_3O_4 film on top of MgO is patterned with the shape shown in [Fig. 2.1b](#)—left. The processes involved are:

- a. A thin film of Fe_3O_4 is epitaxially deposited on MgO (001) by Pulsed Laser Deposition. This work is part of the thesis of Orna [1].
- b. The Fe_3O_4 thin film is spin coated with a photoresist. This is a polymeric photosensitive material that can be spun onto the wafer in liquid form. The spinning speed and photoresist viscosity will determine the final resist thickness. For the first step, we used a typical thickness of $2.4 \mu\text{m}$.
- c. The substrate is soft-baked during 50 s at 110°C on a hot plate, in order to remove the solvents from the resist, minimize the stress, and improve the adhesion.

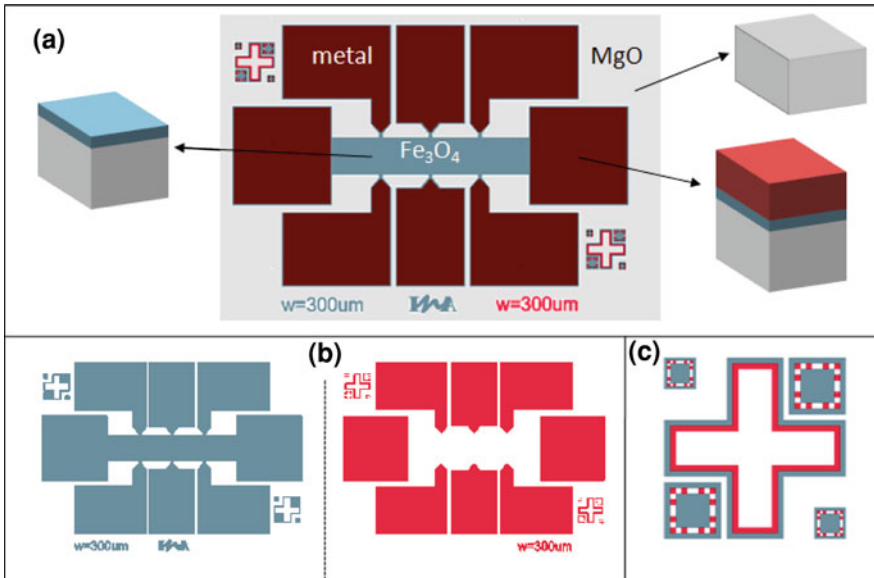


Fig. 2.1 Lithography mask designed by computer. **a** Top and 3D view of the mask. The red, blue and grey symbols symbolize a metallic material, Fe_3O_4 and the substrate MgO, respectively. **b** The two patterns included in the design. Each pattern requires a separated complete lithography process. **c** Marks used, which are necessary for the good alignment of the two steps

- d. The mask is aligned to the wafer using a *Karl Suss*[®] mask aligner, and the photoresist is exposed to a UV source, passing through the photomask (Hg light source, h-line: $\lambda = 405 \text{ nm}$) during 7 s. The areas of the mask with chromium are completely opaque to the light and complementary areas are transparent. The light passes through the mask. The zones of photoresist which are exposed to light undergo a chemical reaction upon this exposure. As the resist used is positive, exposed zones become more soluble in the developer used.
- e. The resist is developed, leaving regions that are covered by photoresist and complementary regions that are not covered (as the resist is positive, it takes the shape of the structure designed, the blue structure in Fig. 2.1b—left).
- f. The remaining resist is hard baked (125°C , 2 min on a hot plate) to harden it for next processes.
- g. The sample is introduced into a *Sistec*[®] ion etching equipment, where $\sim 300 \text{ eV}$ argon ions impinge on the sample, etching it. As the Fe_3O_4 films have a maximum thickness of 40 nm, unprotected parts are removed, whereas those which have resist on top remain unharmed by this process. By the use of an electron neutralizer gun, the ions are neutralized ($\text{Ar}^+ \rightarrow \text{Ar}^0$) before arriving at the sample, avoiding charging effects at the surface of the insulator MgO substrate, which would eventually spoil the etching process after a certain point.
- h. The remaining resist is removed by acetone immersion.

Second step: Metallic pads are deposited on top of the Fe_3O_4 , with the shape of Fig. 2.1b—right. The processes are as follows:

- a. The sample is spin coated with an image-reversal (negative) photoresist. The thickness chosen was of 3 μm .
- b. The resist is soft-baked during 2 min at 100°C on a hot plate.
- c. The mask is aligned to the wafer, using a mask aligner, and the photoresist is exposed ($t = 10$ s). In principle, the exposed zones become more soluble. Marks shown in Fig. 2.1c are used for a good alignment of both structures.
- d. A reversal bake is done, at 130 °C during 2 min. This process crosslinks the exposed photoresist, becoming insoluble in the developer. The unexposed areas still behave like a normal unexposed positive resist.
- e. A flood exposure, without mask, is done (27 s). Thus, the zones which were first exposed (step c) are much more insoluble in comparison with those which were not.
- f. The resist is developed, leaving regions in the sample that are covered by photoresist and complementary regions that are not covered (as the resist is negative, resist forms the complementary structure of that designed, Fig. 2.2b—right).
- g. The sample is introduced into an *Edwards*® electron-gun evaporator. A 100 nm thick metal layer (typically polycrystalline gold or aluminum) is evaporated. Usually a thin layer (around 10 nm) of chromium is deposited beforehand, for good adhesion of the metallic layer.
- h. The sample is immersed in acetone. The profile of the negative resist, in contrast to the positive one (see in detail the resist profiles in Fig. 2.2), favors the complete removal of those parts of metal with resist behind them. This type of process is called “lift-off”.

An optical image of the sample, with gold as metallic layer, is shown in Fig. 2.3a. We should remark that MgO substrates become cracked when immersed in an ultrasound bath. This makes this lithography process a bit more complicated than when using, for example, normal silicon substrates.

The pads are finally connected by ultrasonic wire bonding to a chip carrier, with a *Kulicke & Soffa Ltd*® instrument. The chip carrier (Fig. 2.3b) has macroscopic connections, which can be manipulated in a conventional way.

The lift-off process explained has been used in other types of designs for applications using the Dual Beam system (Sect. 2.1.2). In this case, metallic circuits are patterned by photolithography, consequently nano-lithographed by the Dual Beam equipment. The substrate chosen for these applications is a silicon wafer, with a 150–250 nm thick insulator on top of it (either Si_3N_4 deposited by Plasma-Enhanced-Chemical-Vapor-Deposition, using *Sistec*® PECVD equipment, or thermally oxidized SiO_2). This layer insulates electrically the metallic pads and, at the same time, electrons or ions can be partially evacuated thorough the Si substrate, avoiding charging effects which ruin the nanolithography process.

An image of the equipment used for the photolithography is shown in Fig. 2.4. The minimum line width obtained routinely in our laboratory in these processes is

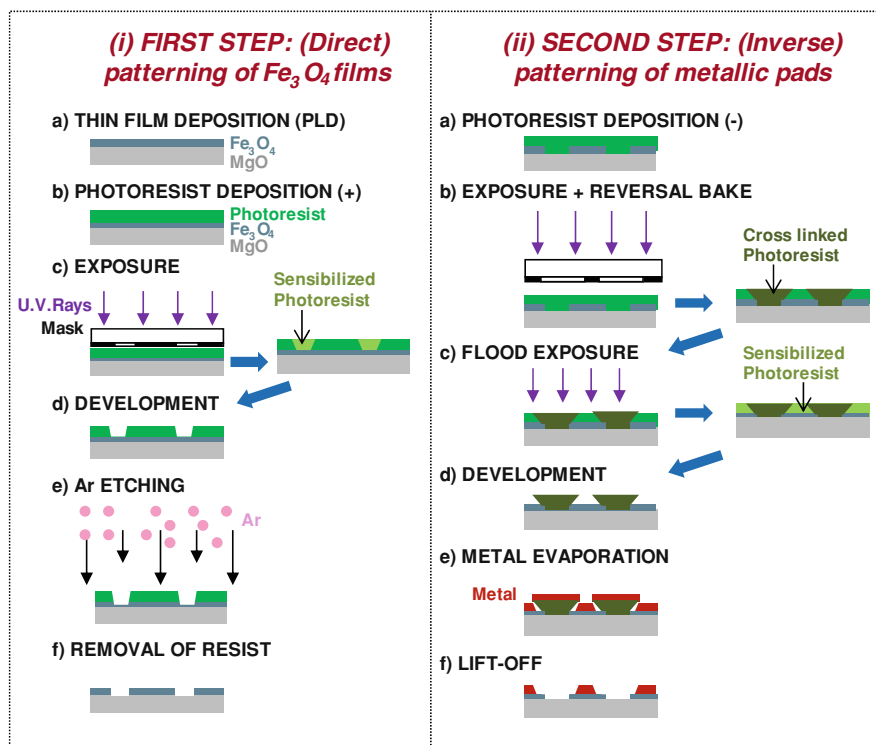


Fig. 2.2 Photolithography of Fe_3O_4 thin films. The two layers are patterned in two separate processes. In this scheme the baking steps, explained in the text, are omitted. The photoresist profiles are exaggerated for pedagogical reasons

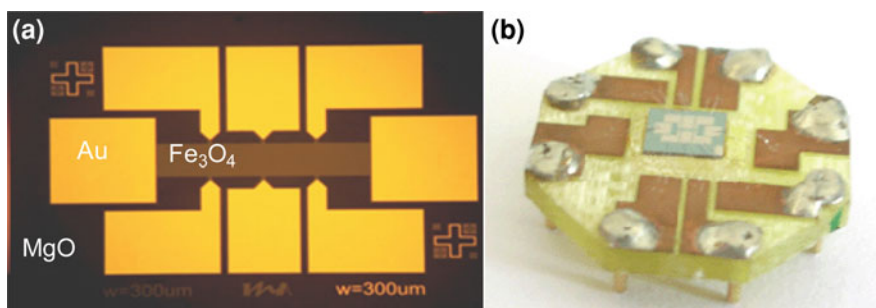


Fig. 2.3 **a** Optical image of one Fe_3O_4 bar with gold pads on top, the result of a two-step lithography process. **b** Sample mounted onto a chip carrier and microcontacted. The chip carrier was designed together with the Dr. Óscar Montero and the technician Carlos Martín

around 3–4 μm (better in the case of positive resist). The final resolution in an optical process is given by the diffraction limit ($\lambda/2$, λ being the light wavelength), although other effects such as optical aberrations of the system, the exposure mode

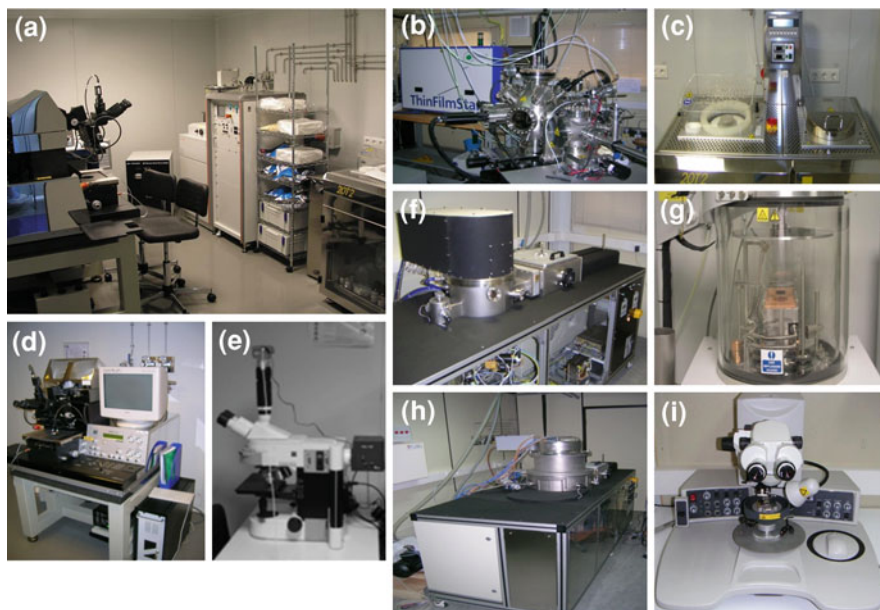


Fig. 2.4 Images of the instruments used for photolithography. **a** General view of the clean room. **b** Pulsed-Laser-Deposition equipment. **c** Spin coater and hot plate. **d** Mask aligner. **e** Optical microscope. **f** Plasma-Enhanced-Chemical-Vapor-Deposition equipment. **g** Electron-gun evaporator. **h** Ion milling equipment. **i** Wire bonding equipment

and the chemical process linked to the resistance are what actually dictate the limits. In the semiconductor industry, critical dimensions below 100 nm are nowadays routinely patterned in integrated circuits [2, 3]. Huge steppers working in projection mode, KrF or ArF lasers and immersion lenses to increase the numerical aperture are used to push the limits of this technology [2, 3]. For a more detailed explanation of these processes, as well as of general aspects of lithography, see Ref. [4, 5].

2.1.2 Dual Beam System

A wide description of a Dual Beam, integrating a SEM and a FIB column for nanolithography, was given in the introductory chapter, due to its relevance on the thesis and novel character of the processes performed. In general, some of the main applications of these systems are [6]:

1. FIB etching and simultaneous SEM imaging.
2. Imaging with both beams: although the electron beam is the primary imaging tool in a dual beam, the imaging from the ion and electron beams is often complementary. The collection of secondary ions is possible when using FIB.

3. Cross-section fabrication and analysis.
4. Local deposition of materials by introducing precursor gases inside the chamber (FEBID and FIBID, [Sect. 1.5.3](#)).
5. Lamella preparation for transmission electron microscopy (TEM) analysis. Thin specimens, transparent to high-energy electrons can be fabricated. Before the etching, a protective layer is deposited. After several progressive thinning processes, a micromanipulator takes the lamella to the TEM grid. This method is becoming popular in microscopy laboratories as an alternative method to the manual methods.

In [Fig. 2.5](#) images of the *Nova Nanolab 200* by FEI® experimental system are shown, including columns, detectors, manipulator, and needles for gas injection (GIS). The processes are carried out in a chamber at high vacuum (turbo-molecular pump), whereas both columns need ultra-high vacuum (ionic pumps).

As commented in [Sect. 1.5](#) this system has been used to create nanostructures:

- By simultaneous FIB etching and electrical resistance control, we have created atomic-sized nanoconstrictions, using the SEM column for imaging the process.
- By the injection of a gas precursor using the GIS, we have deposited nanowires of different materials using both focused columns (FEBID and FIBID).

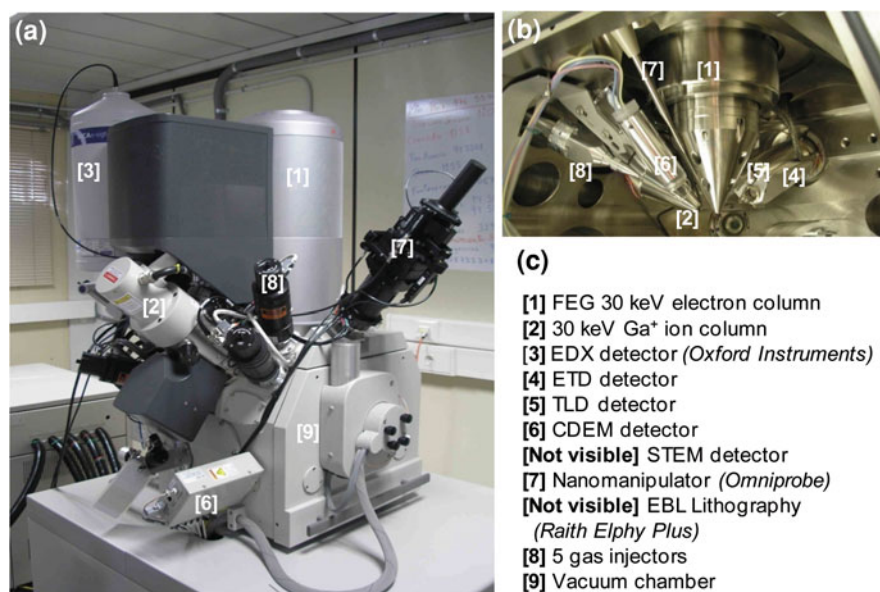


Fig. 2.5 Dual Beam equipment used. **a** General overview of the system. **b** View inside the chamber **c** Names corresponding to the numbers indicated in (a) and (b)

2.2 Electrical Measurements

2.2.1 Magnetotransport Measurements as a Function of Temperature

For the study of the electrical transport measurements of samples, as a function of temperature and magnetic field, we have used a combination of several items of equipment belonging to the ICMA (illustrated in Fig. 2.6):

- Combined Keithley[®] system, composed of a 6220 DC current source, and a 2182A nanovoltmeter. By injecting a constant current (DC), the voltage of the device under test is measured. A wide range of resistances can be measured, from 10 n Ω to 1 G Ω .
- Closed-Cycle-Refrigerator (CCR) by Cryocon[®]. By means of the thermodynamic cycle in He gas, the cryostat can lower the temperature of the sample down to 25 K. The control of temperature is performed by standard PID parameters.
- Electromagnet by Walker Scientific[®], delivering maximum magnetic fields of 11 kOe.

A scheme of the system is shown in Fig. 2.7. The control of all equipment via PC was done by the design of Labview[®] programs. All this work was carried out jointly with Dr. Jan Michalik.

The electrical measurements are normally done in a 4-probe configuration, to avoid to measure contact resistances in the measurements. This geometry is schematized in Fig. 2.7.

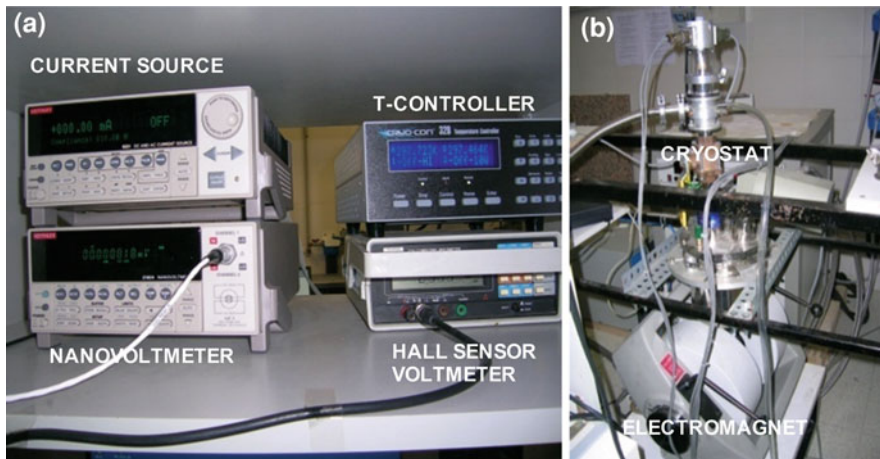


Fig. 2.6 Images of the system used for magnetotransport measurements as a function of temperature. **a** Equipments for electrical measurements, as well as for the control of temperature and magnetic field. **b** General view of the system

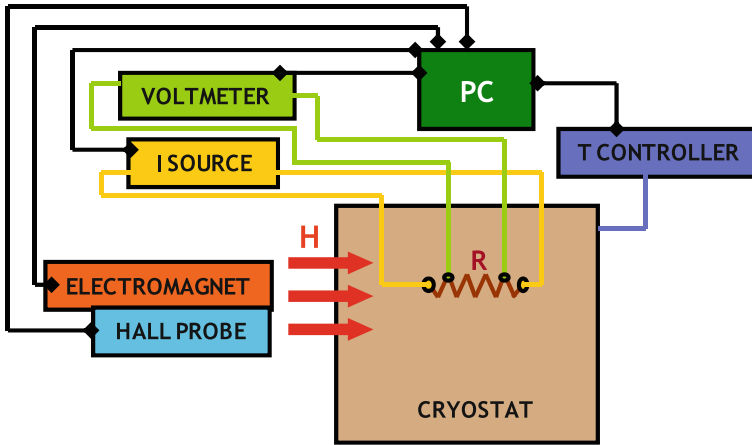


Fig. 2.7 Scheme of the system for magnetotransport measurements as a function of temperature

The Keithley system has been used in two modes of measurements:

- *Normal DC mode*: A constant current is applied, and voltage is measured.
- *Delta mode*: The voltage is measured with alternating positive and negative test current. This allows the cancelling of constant thermoelectric voltages by alternating the test current. It significantly reduces white noise, resulting in more accurate low resistance measurements when it is necessary to apply very low power. A few electrical measurements for this thesis were done in a commercial Physical Properties Measurement System (PPMS) from Quantum Design[®], situated in the Instrumentation Service of the University of Zaragoza. This was used when high magnetic fields (maximum field of 9 T) or low temperatures (minimum temperature of 300 mK) were necessary, for resistances below $\sim 1 \text{ M}\Omega$.

2.2.2 “In Situ” Electrical Measurements

Some of the results obtained in the thesis have been achieved by a combination of the nanolithography techniques with electrical measurements. The usual approach followed consists of the study of the devices after its creation or modification. However, in this case we have also sometimes carried out simultaneous electrical measurements while the nanostructure was being patterned. To do this, two types of special stages were incorporated into the Dual Beam system (see Fig. 2.8):

1. Four Kleindiek[®] electrical microprobes, separately moved by a motor or piezoelectric, till contacted to pads. They are made of tungsten, and have a final diameter of around $1 \mu\text{m}$.

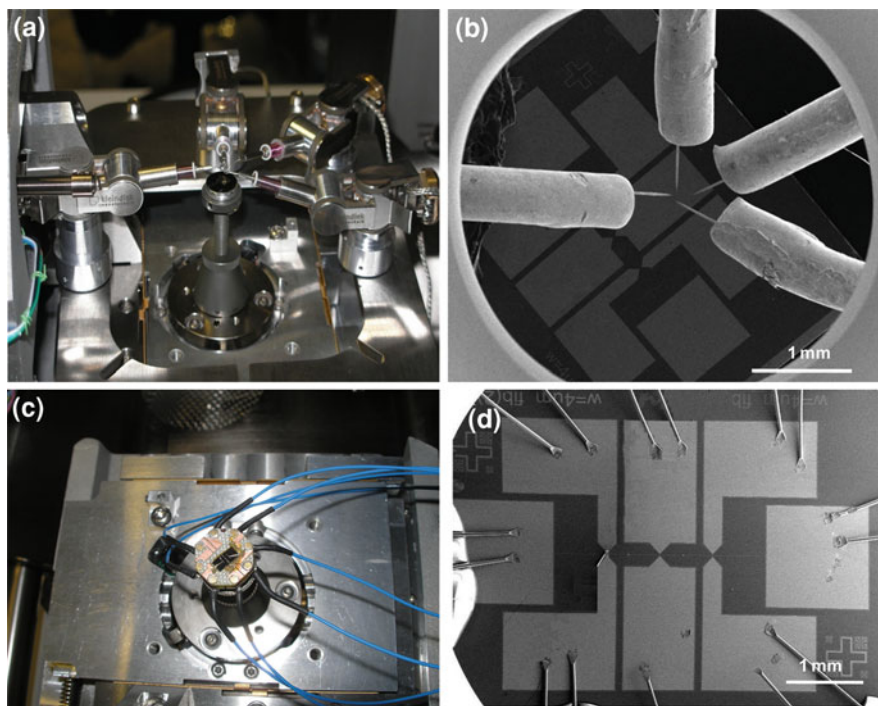


Fig. 2.8 Images of methods for electrical measurements inside the Dual Beam chamber. **a** Electrical microprobes stage. **b** SEM image of the microprobes inside the chamber, with a sample in a plane below. **c** Stage for electrical measurements. A sample is mounted in the image. **d** SEM image of a sample with microcontacts connected to the stage

2. Home-made stage, with eight connections, with the sample contacted by wire-bonding to a chip carrier, loaded into the stage.

The electrical wires are transferred outside the chamber by a port, and connected to the same Keithley system as explained before.

2.3 Spectroscopic Techniques

We have mainly used two different spectroscopy techniques to characterize the composition and chemical nature of the samples studied: energy dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscopy (XPS).

2.3.1 Energy Dispersive X-Ray Spectroscopy

We showed in Fig. 1.11a, that as a consequence of the interaction of electrons with matter in the keV range, X-ray photons are emitted. The PE produce the emission

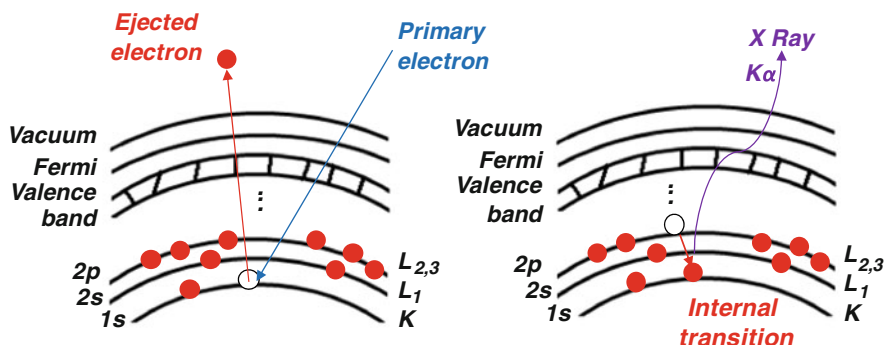


Fig. 2.9 Schematic diagram of the EDX process. Relaxation of the ionized atom results in photon radiation, characteristic for each element

of inner electrons, which create a hole in an atomic level. An electron from an outer, higher-energy shell can fill that hole, emitting an X-ray, with a characteristic energy equal to the difference between the higher and the lower energy shell. As the energy of the X-rays is characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured. See a scheme of the process in Fig. 2.9.

An Oxford Instruments[®] EDX was used, incorporated into the Dual Beam system, for in situ compositional characterization (see Fig. 2.5). The interaction volume of the electron beam with the sample determines both the lateral resolution and the depth of analysis; this is a function of the primary beam energy, invariably of the order of one micrometer. The energy resolution is ~ 150 eV. This permits a compositional quantification to be made of the sample probed.

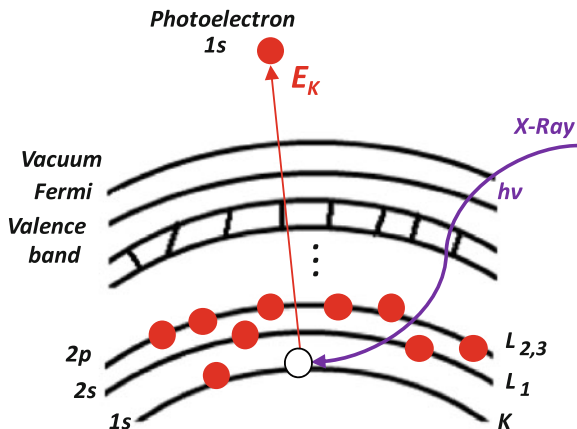
2.3.2 X-Ray Photoelectron Spectroscopy

XPS (traditionally called ESCA) is a spectroscopic technique based on the photoelectric effect, i.e., the ejection of an electron from a core level by an X-ray photon of energy $h\nu$. The energy of the emitted photoelectrons is then analyzed by an electron spectrometer. The kinetic energy (K) of the electron is the experimental quantity measured by the spectrometer, but this value will depend on the X-rays energy. The binding energy of the electron (BE) is the parameter which identifies an element specifically. The equation which describes the process is

$$BE = h\nu - K - W \quad (2.1)$$

where W is the spectrometer work function. Albert Einstein received the Nobel Prize in Physics in 1921 for his interpretation of the photoelectric effect. Figure 2.10 shows a scheme of the XPS process.

Fig. 2.10 Schematic diagram of the XPS process, showing the photoionization of an atom by the ejection of a 1 s electron



For these experiments we used a Kratos[®] Axis Ultra DLD equipment located at the INA. A monochromatic Al $K\alpha$ X-ray source was used, with exciting energy $h\nu = 1486.6$ eV. The sampling depth of the X-ray is at the micrometric level, while only those photoelectrons from the outermost, ~ 10 nm or less, escape without energy loss. These are the electrons used for quantitative quantification; XPS is thus a surface-sensitive technique. An in-depth study can be performed, since the equipment incorporates a 5 kV - Ar^+ gun, which progressively etches layers of material, which are consequently probed by XPS. A hemispherical detector analyses the electron energy. The experiments require ultra-high vacuum conditions (ionic pump).

Due to the difficulty in focusing X-rays, the effective minimum area probed in the equipment is approximately $30 \times 30 \mu\text{m}^2$. The high-resolution of the equipment (maximum = 0.2 eV) permits the valence state of the atoms composing the sample to be distinguished.

2.4 Spatially Resolved MOKE Magnetometry

The main magnetometry technique used in the thesis is a magneto-optic technique: the spatially resolved Longitudinal Magneto-Optical-Kerr-Effect (MOKE). These measurements were done at Imperial College, London, in the research group of Prof. Cowburn.

The Kerr effect consists of the rotation of the plane of polarization of a light beam when reflected from a magnetized sample. We show in Fig. 2.11. the geometry for the longitudinal Kerr-effect (L-MOKE). Radiation from a light source is first passed through a polarizer. The resulting plane-polarized light is then incident on a sample. The L-MOKE is sensitive to the in-plane magnetization component of the sample, and thus it is in the incidence plane of light. The magnetization changes the angle of polarization of light, as well as inducing an

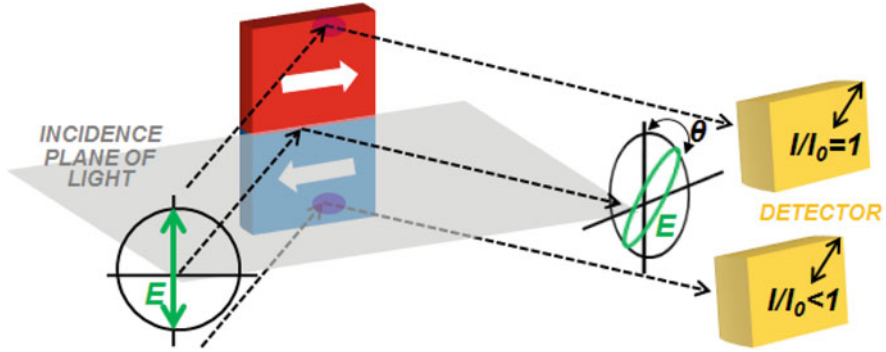


Fig. 2.11 Geometry for the Longitudinal-MOKE (see text). In the central ray of light, the polarization of the electric field changes from linear to elliptical polarization, with a change in the angle of polarization, θ . In the top ray, the detector is aligned to make the signal maximum. Thus, due to the contrary angle rotation in the bottom ray, resulting from an opposite magnetization direction in the sample, the signal collected by the detector is smaller

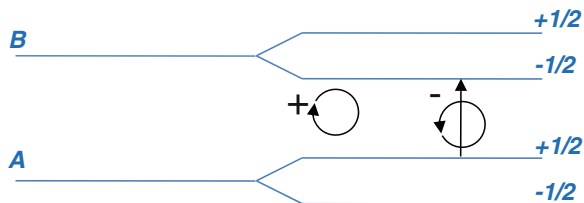
ellipticity in the initially linearly polarized light. The thickness probed is typically of 20 nm. In the example of Fig. 2.11, the sample contains two domains magnetized in opposite directions. The light incident on one domain is rotated in the opposite direction from that incident on the other domain. Therefore, if the analyzer is oriented such that the light from the first domain is maximum, then the plane of polarization of the light reflected from the other domain is not aligned with the analyzer, and the signal is reduced.

A simple explanation for this effect is as follows [7]. Linearly polarized light can be decomposed in two oppositely circular polarizations. The angular number of both is equal to 1, but in the right circular polarization (+), $m_L = +1$, whereas the left circular light (−) has $m_L = -1$. Using the simple example of a magnetic material with atomic spin $S = 1/2$, the exchange interaction splits its energy levels into two sub-levels, with total spin $m_S = +1/2$ and $m_S = -1/2$.

Both energy and angular momentum must be conserved when a photon excites an electron from one sub-level in A to one in B. Selection rules dictate that $\Delta m_L = \pm 1$, implying that only transitions drawn in Fig. 2.12 are possible. Thus, oppositely polarized photons correspond to different electronic transitions in the atom. If the electronic population in B sub-levels differ one from the other, the absorption of one polarization is greater than the other (a phenomenon called circular dichroism). When the resulting circular polarizations are recombined again, the plane of polarization is rotated with respect the incoming beam. The resulting phase difference between the initial and final planes of polarization is called circular birefringence.

The magneto-optical effects do not directly provide absolute values for the magnetization. The MOKE rotation depends on the angle of measurement and wavelength of light, as well as on the magneto-optic constant, which is material dependent at a determined temperature. Thus, MOKE measurements provide

Fig. 2.12 Absorption of light in a ferromagnet with $S = 1/2$



hysteresis loops in intensity (arbitrary units) as a function of the magnetic field. The Kerr effect is normally used for measurements of hysteresis loops in magnetic thin film layers, or for the imaging of magnetic domains. However, this experimental setup [8, 9], now commercialized by Durham Magneto Optics[®], is suitable for the measurement of magnetic nanometric structures. A diode-pumped solid state laser ($\lambda = 532$ nm) with a diameter of about $5\text{ }\mu\text{m}$ (FWHM) is used as a probe. A CCD camera is used for pre-alignment of the nanometric structure, located on a substrate. The final alignment is done by performing a reflectivity map of the zone of interest. A motor permits the stage to be moved with a resolution of 50 nm . A quadrupole is used for applying AC magnetic fields ($1\text{--}27\text{ Hz}$), with $H_{\text{max}} \sim 400\text{ Oe}$. The system is a state-of-the-art-MOKE instrument having, for instance, a sensitivity for Permalloy $S = 6 \times 10^{-12}\text{ emu}$ at room temperature [9] (in a SQUID magnetometer $S \sim 10^{-8}\text{--}10^{-9}\text{ emu}$). This high sensitivity permits the measurement of hysteresis loops in sub-micrometric and sub- λ nanomagnets. As the diameter of the laser is bigger than at least one of the dimensions of the nanostructure probed, a dilution factor is present in the MOKE signal with respect to the bulk material.

2.5 Atomic Force Microscopy

In the previous sections we have cited two main microscopy techniques used for this work: the optical microscope, allowing the observation of structures with sizes slightly below $1\text{ }\mu\text{m}$, and the scanning electron microscope, which can resolve objects around 1 nm in size.

In this section, we will comment on some aspects of another microscope used, with resolution of fractions of a nanometer: the atomic force microscope (AFM). AFM is a type of scanning probe microscope (as is its parent, the scanning tunneling microscope), based on the interaction of a sharp tip with the surface sample when it is brought into proximity to the surface. The tip is the end of a microscopic cantilever, used to scan the specimen surface. The cantilever is typically of silicon or silicon nitride with a tip radius of curvature in the order of nanometers. The forces between the tip and the sample lead to a deflection of the cantilever, which can be modeled with Hooke's law. The typical AFM forces are mechanical contact force, Van der Waals force, capillary forces, electrostatic forces, magnetic forces

(MFM), etc. Typically, the deflection is measured using a laser spot reflected from the top surface of the cantilever into an array of photodiodes.

The AFM images were taken at Imperial College, London, with a Veeco Multimode[®] AFM. The Ph.D student Liam O'Brien made a part of the measurements. Contact mode was used for imaging, where the force between the tip and the surface is kept constant during scanning by maintaining a constant deflection. The tip deflection is used as a feedback signal, which is used to form an image of the structure probed.

Some clear advantages of AFM compared to SEM can be mentioned. There is better spatial resolution, the measurements do not require vacuum conditions, a true three-dimensional surface profile is obtained, and there are no charging-effect problems with insulator samples. However, there are also important disadvantages of AFM compared with SEM. The SEM can image an area on the order of mm $\times$ mm, with a depth of field in the order of mm. The AFM can only image a maximum height in the order of microns, and a maximum scanning area of a few μm^2 . Besides, image artifacts are common, especially if an incorrect choice of tip for the required resolution is made. The significant lower speed in the image is also an important drawback, as well as the possible hysteresis in the piezoelectric. We should mention for the sake of completeness that AFM is also used as a tool for nano-patterning, by local oxidation, dip-pen nanolithography, etc.

2.6 High Static Magnetic Fields

The high static magnetic field experiments were performed at the High Field Magnet Laboratory (HFML) of the University of Nijmegen, Netherlands. The high fields are generated by a Bitter type magnet, consisting on stacked copper (no magnetic material) disks, with very high current densities flowing in them. Small vertically aligned holes are pierced to allow cooling by a high pressure water flow (see Fig. 2.13a). Static fields as high as 33 T can be generated at maximum power of 20 MW (40 kA at 500 V). This is the technique nowadays that produces the highest static magnetic fields (we can compare it with the maximum field produced by: a solenoid without cooling ~ 0.1 T, an electromagnet $\sim 2\text{--}3$ T, or a superconducting Nb₃Sn coil at 4.2 K ~ 20 T [10]). Fig. 2.13b shows an experimental station of the Laboratory, where the coils are inside a cryostat for measurements as a function of temperature. Dr. Uli Zeitler and Erik Kampert were our local contacts for these experiments.

2.7 Other Techniques

Some results in this thesis will be presented with other techniques different from those previously explained, which should also be mentioned. The person in charge

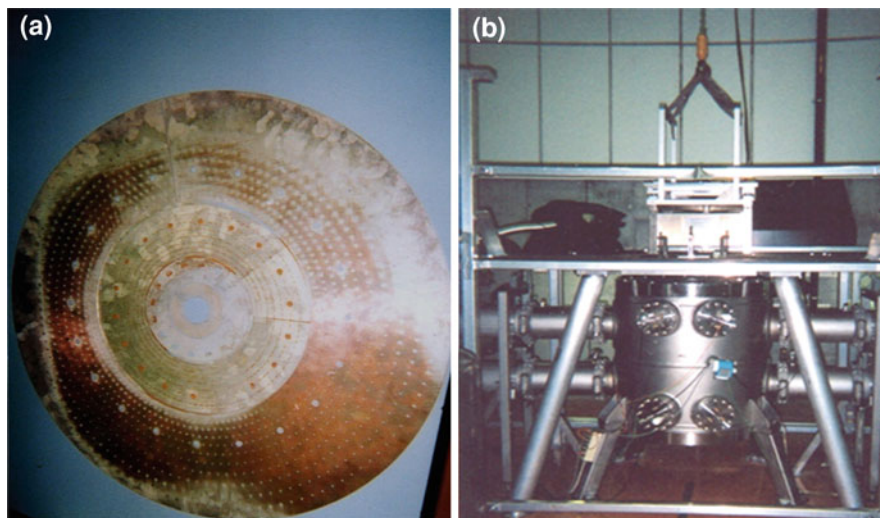


Fig. 2.13 a Bitter coil. b Experimental station at the HFML

of the measurements is cited in the text when a particular result is presented in the following chapters.

Pulsed Laser Deposition, PLD (INA): Fe_3O_4 thin films were epitaxially grown on MgO (001) substrates. A KrF pulsed laser produces an ablation in a Fe_3O_4 target, depositing stoichiometrically the iron oxide on the substrate.

High-Resolution X-ray Diffraction, HR-XRD (INA): structural characterization of thin films. Determination of the degree of crystallinity of a film grown on a substrate.

Superconducting quantum interference device, SQUID magnetometer (Scientific Services of University of Zaragoza): a constant current passes through two in-parallel Josephson junctions. The applied voltage oscillates with the changes in the phase of the two junctions, which depend upon the change in magnetic flux. This results in a highly sensitive magnetometer.

High-Resolution Transmission Electron Microscope, HRTEM (Scientific Services of the University of Barcelona, CEMES-Toulouse): 200–300 kV electrons are accelerated, being transmitted through a thin sample (~ 100 nm). The image formed, a consequence of the diffraction of electrons with the material, can be transformed to the real space, resulting in an image of the crystallographic structure of a sample at an atomic scale.

Scanning-Tunneling-Spectroscopy, STS (University Autónoma of Madrid, group of Prof. Sebastián Vieira): the local density of electronic states of a surface is probed at an atomic scale, by measuring the tunneling differential conductance between a STM tip and a sample at low temperatures.

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