Magnetic imaging and microscopy

Electron Microscopies

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This lecture will present some techniques linked to electron micros copies to measure locally the magnetic properties of magnetic materials. The development of local measurement tools is necessary as the dimension of the devices shrink down.
The scanning electron microscope (SEM) gives information from the surface of the sample while the transmission electron microscopy is useful to get information from the volume (even if TEM gives 2D information projected along the beam direction). TEM is also a useful tool to correlate structural and magnetic properties and get information on the chemistry of the samples.

This lecture will deal with one technique based on SEM: the SEMPA (Scanning Electron microscopy with Polarised Analysis) technique and two techniques based on TEM : the Lorentz microscopy and the electron holography. For each technique, I will describe the theoretical principle, the experimental set-up, some applications and some remarks on sample preparation and resolution. This lecture is not an exhaustive list of all the things that can be done but a short review of the examples published by different groups. Before giving all these details on the techniques, I will introduce the two types of microscopes involved in the techniques.

General description of electron microscopes

On this schematic are drawn 3 types of microscopes to make analogy between the different techniques. They all consist in sources (filament or field-emission gun), lenses (electromagnetic lenses to deviate the electron beam) and detectors (fluorescent screen, electron detector). The electrons need to propagate freely in the microscope; vacuum is therefore required in the microscopes.

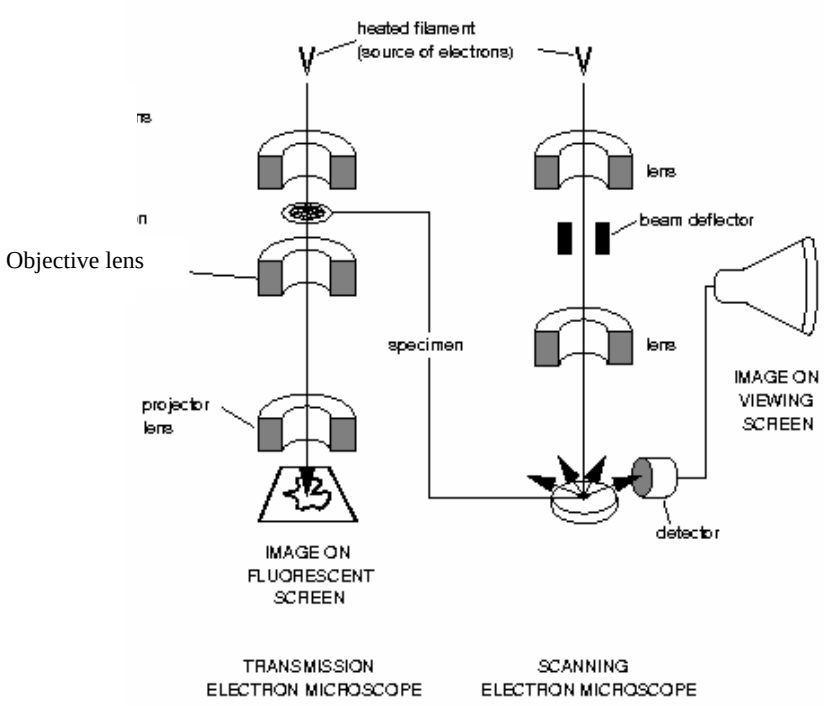

Image from http://www.mih.unibas.ch/Booklet/Lecture/Chapter1/Fig.1-6.gif

Scanning Electron Microscope	Transmission Electron Microscope
Voltage : 5keV-30keV *Spatial resolution* : around 50Å *Some analysis* : Secondary electrons : topography, backscattered electrons : atomic number contrast, X-ray : chemistry	*Voltage* : 80keV-400keV *Spatial resolution* : lower than 2Å *Some analysis* : transmitted electrons (elastically and inelastically: EELS), X-ray (EDX)

SEMPA

Scanning Electron microscopy with Polarised Analysis

- Theoretical principle

SEMPA measures the spin polarization of the secondary electrons that exit from a magnetic sample as the finely focused (unpolarized) beam of the scanning electron microscope rasters over the sample.
These secondary electrons are predominantly produced by the interactions between energetic beam electrons and weakly bonded conduction-band electrons in metals. The secondary electrons are sensitive to topography, the total secondary electron current measured gives topographic information. If the sample is ferromagnetic, the emitted electrons are spin-polarized due to the difference of occupancy of the up and down bands.
The polarization is measured using a Mott detector and a Wien filter, giving access to the polarisation in 3 dimensions. We will show that the polarisation is linked to the magnetisation.

- Experimental setup

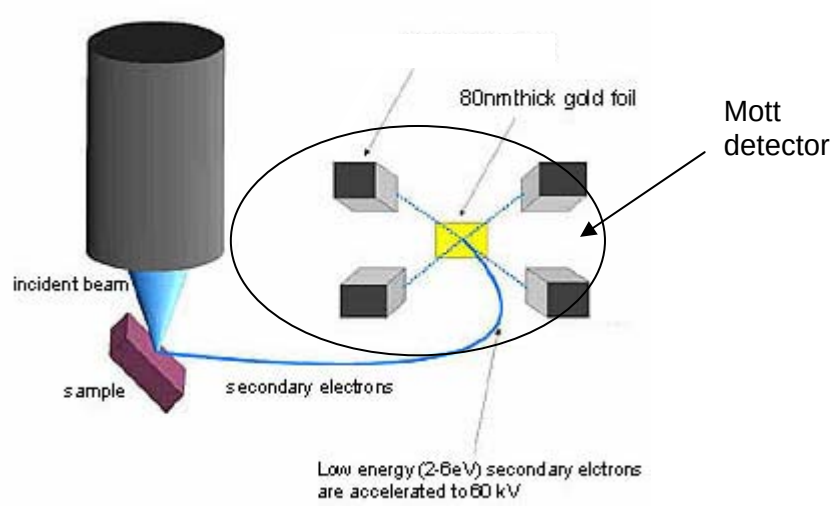

Figure 1 : schematic of a SEMPA set up (http://www.solid.phys.ethz.ch/pescia/sempa.htm)

- Examples

SEMPA has been used since mid eighties on ferromagnetic materials [1]. Since then, many studies have been led on continuous films [2], zigzag patterned elements [3] or nanorings [4]. Some results will be presented in this lecture.

- Spatial resolution

50 nm for LaB_6 guns, 10nm for FEG

- Samples

As SEMPA uses electrons, you have to use conductive samples to get rid of charge problems (or you need to coat your sample with carbon). No specific sample preparation is required.

- Remarks

Measure of the magnetisation
No applied field
Surface technique (secondary electrons are emitted from a depth of 1nm)

Lorentz microscopy

In a ***Transmission Electron Microscope*** or a ***Scanning Transmission Electron Microscope***

- Theoretical principle

Electron moving through a region of space with an electrostatic field and a magnetic field B experiences the Lorentz force F_L : $\mathbf{F}_L = -e(\mathbf{E}+\mathbf{v}\wedge\mathbf{B})$. F_L acts normal to the travel direction of the electron, a deflection will occur. Only the in-plane magnetic B induction will contribute to the deflection. The deflection angle is linked to the in-plane magnetisation and the thickness of the sample.
There are two modes in Lorentz microscopy: the Fresnel mode, in which you observe domain walls and magnetisation ripples, and the Foucault mode, where domains are imaged.
The *Fresnel* mode: in conventional TEM, the minimum observed contrast indicates the in-focus position of the sample. In this position, the domain walls do not appear. In the Fresnel mode, underfocusing or overfocusing is used to image the walls (see figure 2 below)

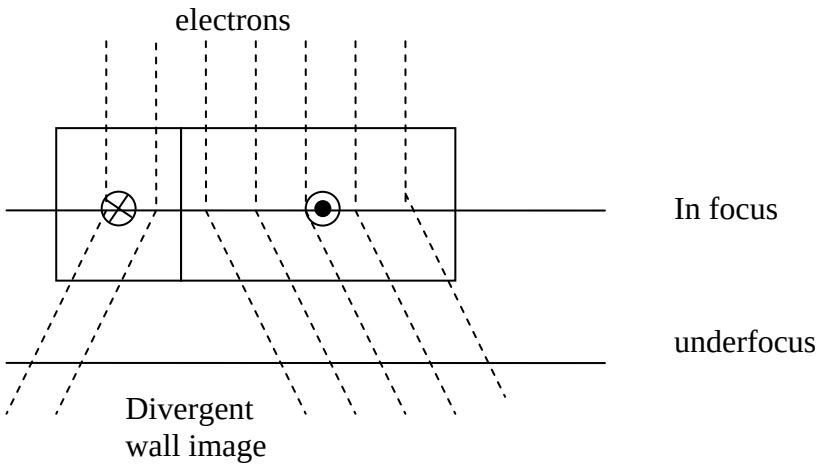

Figure 2 : Schematic of the electron path through a magnetic sample in Fresnel mode

A wall appears as a dark line when there is a deficit of electrons, whereas it is white when there is an excess of electrons. It is then possible to follow the domain wall motion under the application of an external magnetic field. It gives essentially qualitative information. The *Foucault* mode: this mode corresponds to a bright field image mode in conventional TEM. This means that you select a part of your beam with an aperture located in the back focal plane of your imaging lens. The electrons that pass through the aperture will appear bright whereas the others will appear dark. The deflection angles linked to magnetisation are small and you can not really differentiate them from the transmitted beam, you have to put the aperture quite close to the transmitted beam. Getting nice results in this mode is therefore trickier than getting data from the Fresnel mode. This mode gives also quantitative information with the Differential Phase Contrast [5,6] technique.

- Experimental setup

The sample needs to lie in a field free region to avoid external magnetic field (around 2T in the objective lens), we therefore need to turn off this lens. The trouble is that this lens is responsible for the magnification. Two technical solutions have been proposed: the introduction of a Lorentz lens (below the objective lens) or the design of a special objective lens to make sure no field is seen by the sample.
Another requirement is a special sample holder to apply a field. Some examples will be presented in this lecture.

- Examples

Results will be shown on continuous films [7] and patterned elements [8] using Lorentz experiments. An example will also present the interest of studying the magnetic domains in active devices [9].

- Spatial resolution

Below 10 nm

- Samples

Special specimen preparation is required to get electron transparent membranes. Samples are usually deposited directly on Si_3N_4 membranes (non crystalline substrate)

- Remarks

Observation of the domain walls and magnetisation mapping in domains
Observations with an applied field
Transmission technique, the electron goes through the whole stack. If the stack consists of several magnetic layers, the result is the superposition of the response of the different layers.

Electron holography

In a ***Transmission Electron Microscope***

- Theoretical principle

For electron beams in the energy range of 10^3 to 10^7 eV, the principal electron-matter interaction is the change of electron wavelength associated with the acceleration of the electrons by the electrostatic and magnetic field. The wavelength change leads to a phase change of the wave, relative to the phase of a wave in vacuum. For very thin specimens, it is a good approximation to assume that the phase change is proportional to the integral of the potential and/or the induction through the specimen [10]. In conventional transmission electron microscopy, the intensity of the image does not contain any phase information. The holography technique gives access to this information.
The principle of electron holography is the formation of an interference pattern between the electron wave scattered by the specimen and a reference wave of known form.

- Experimental setup

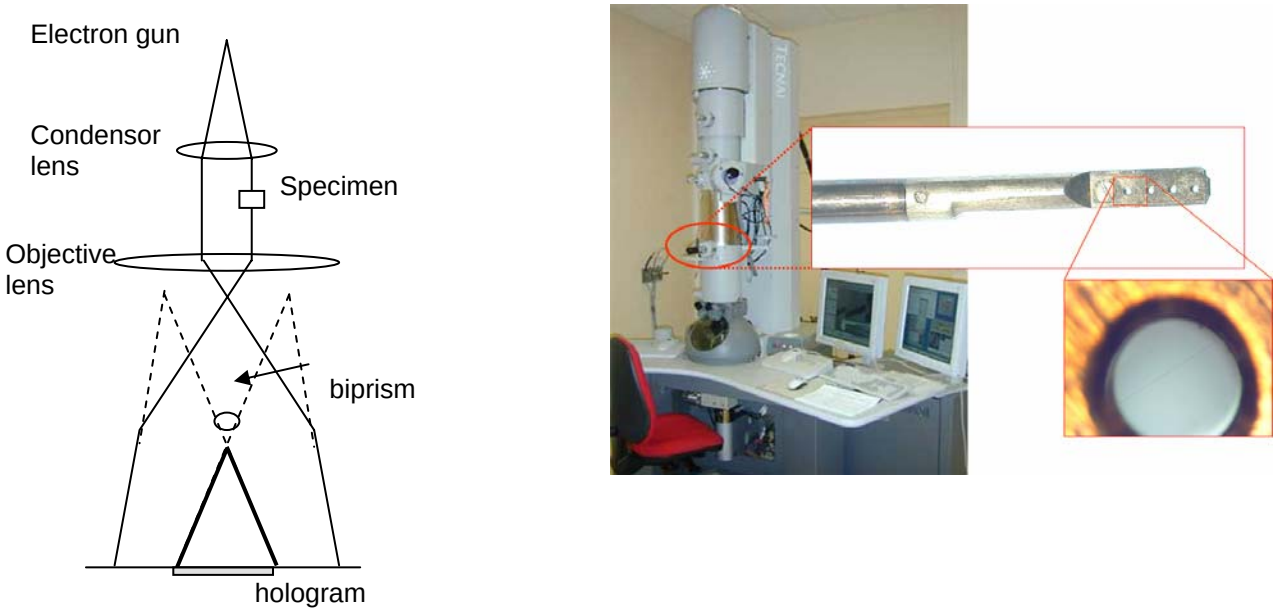

Figure 3 : Left : Schematic of the electron holography set-up (from F.Houdellier website, http://www.cemes.fr/microscopie/) - Right : Photo of the FEI Cs corrected TEM fitted with a FEG, a biprism and a Tridiem (Gatan) – CEMES, Toulouse, France

Holograms are recorded and post-treatments are necessary to extract the phase shift from the holograms. As pointed out in the theory, the phase shift consists of an electric and a magnetic part. Some experimental procedures are required to separate the electric and magnetic parts.
We need to turn off the objective lens as for Lorentz microscopy to avoid the huge objective field. The magnification problem still arises and a Lorentz lens is often fitted into the microscope.

- Examples

First experimental holograms were obtained by Hirayama et al. [11] on barium ferrites. Many studies present results on metal nanowires or nanoparticles [12,13].

- Spatial resolution

Around 5 nanometers

- Samples

As for Lorentz experiments, special specimen preparation is required to get electron transparent membranes. For particles and nanowires, carbon grids are used. Stray fields can also be observed for thick specimens.

- Remarks

Reference holograms need to be recorded and vacuum areas are required close to the region of interest.
CCD cameras are used to record the holograms as their response is linear and the quality of the holograms depends on the recording setup.
Extracting the phase information from holograms needs a lot of numerical processing.

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For details on the TEM, consult the “Introduction to conventional transmission electron microscopy” by M.De Graef, Cambridge University Press, Cambridge (UK), 2003