

PREPARATION AND MAGNETIC CHARACTERIZATION OF Fe/GaAs THIN FILMS FOR XMCD and EMCD EXPERIMENTS

L. Felisari, E. Carlino

**Center for Electron Microscopy, TASC INSTITUTE INFM-CNR
Trieste, Italy**

**F. Maccherozzi, M. Fabrizioli, M. Hochstrasser, G. Panaccione, G. Rossi
APE Beamline, TASC INSTITUTE INFM-CNR Trieste, Italy**

OUTLINE



❖ Motivation and samples description

❖ Sample preparation

GaAs substrate preparation

Fe films growth

❖ Magnetic characterization

Magneto-optic Kerr Effect (MOKE)

X Rays Magnetic Circular Dichroism (XMCD)

Center for
Electron
Microscopy



APE beamline

AIM

Direct comparison between XMCD and Electron Energy Loss Magnetic Circular Dichroism (EMCD)

SYSTEM

Fe thin films on GaAs(001) substrate

Fe

- ❖ well known material system
- ❖ large dichroic signal at $L_{2,3}$ edges

GaAs

- ❖ good lattice match with Fe
(mismatch~1.4%)
- ❖ Well known artifact free TEM sample preparation procedures

GaAs SUBSTRATES PREPARATION

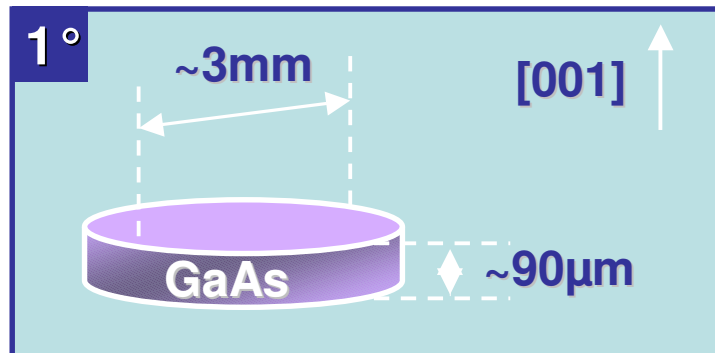


Substrate

GaAs (001)

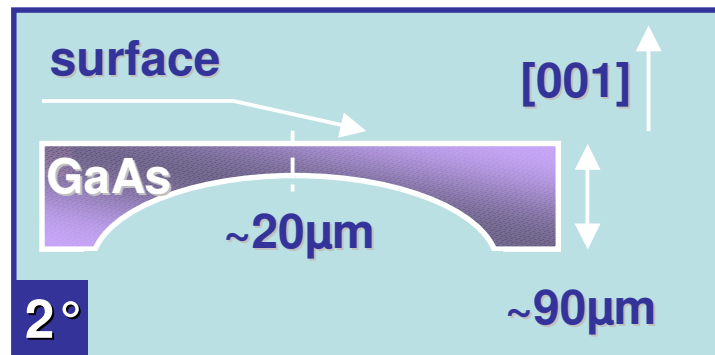
($a_0=0.56\text{nm}$, space group F-43m)

TEM substrate preparation (plan view)



1° step:

3mm diameter disks pre-thinned down to $\sim 90\mu\text{m}$



2° step:

Dimpling and polishing of the central area (final thickness $\sim 20\mu\text{m}$)

Fe growth surface untouched

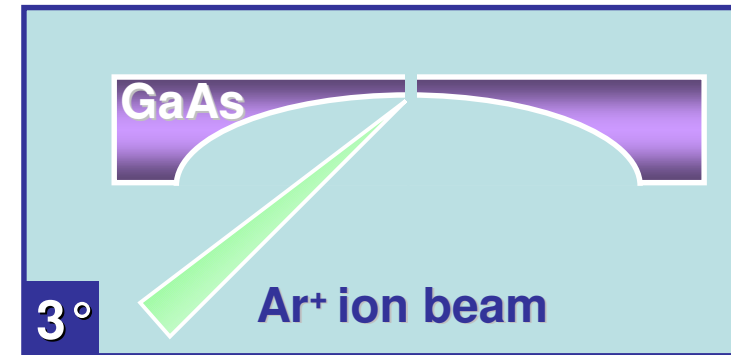
GaAs SUBSTRATES PREPARATION



3° step:

Final thinning by low energy ion milling until electron beam transparency

Ar⁺ ions { energy 4.5÷1.8 KeV
Incidence angle ~ 5°



two options { Ion milling *before* Fe deposition
Ion milling *after* Fe deposition

- ❖ Analysis of influence of GaAs substrate on Fe growth
- ❖ Studies of the influence of ion beam milling on Chiral properties of the samples.

GaAs SURFACE PREPARATION BEFORE Fe GROWTH



(2×10^{-10} mbar)



Ar⁺ ions sputtering (750 eV, sputtering pr. 1×10^{-6} mbar)

Annealing at 580 °C



Substrates' surface analysis

Auger Electron Spectroscopy (AES)

C and O under detection limit (2%)

Traces of Mo

Low Energy Electron Diffraction (LEED) → 6x4 surface reconstruction

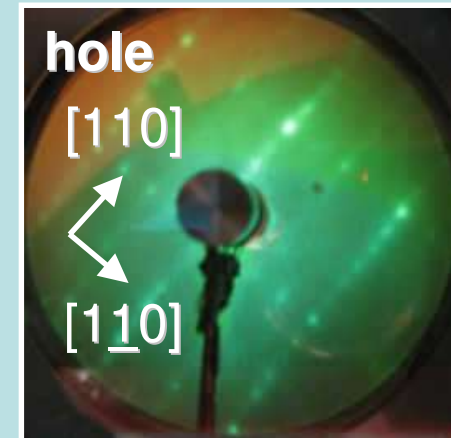
LEED on GaAs surface



Surface quality influences

Fe growth and its

magnetic properties



Better surface quality

Fe GROWTH

Fe (bcc, $a_0=0.286\text{nm}$, space group $Im-3m$)

- ❖ Molecular beam epitaxy (MBE) in UHV chamber
- ❖ Small lattice mismatch ($\sim 1.4\%$)
- ❖ Films thickness $10\div 15\text{nm}$
- ❖ Cu capping



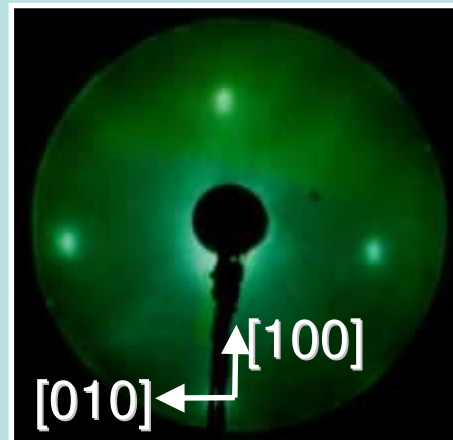
Fe surface analysis

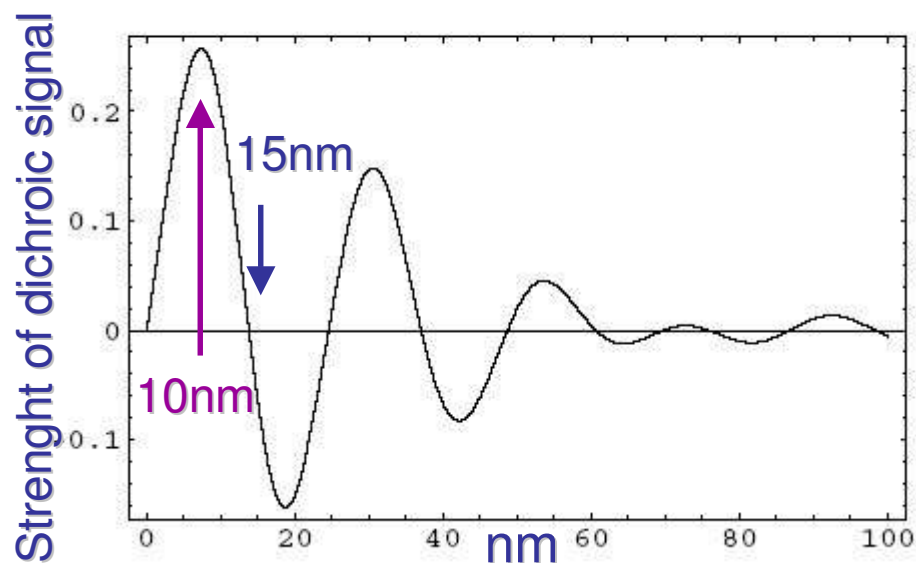
LEED on Fe surface



Good surface quality

1x1 reconstruction





Why 10 and 15 nm?

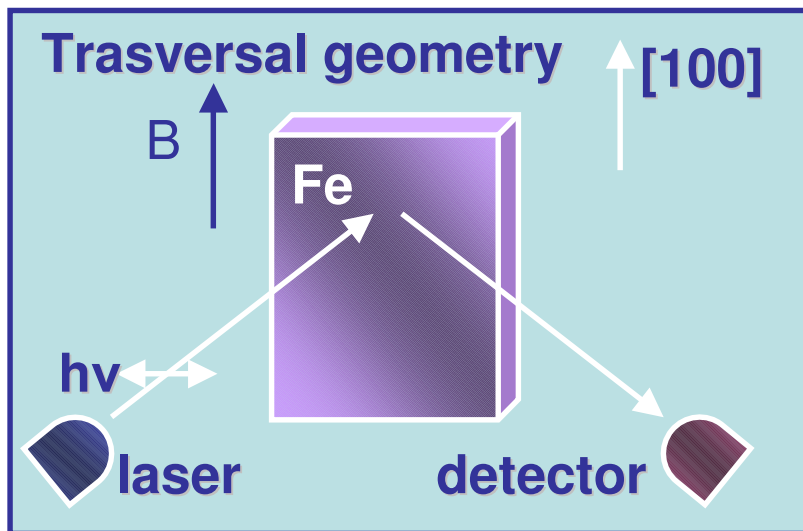
- ❖ 10 nm: maximum dichroic signal
- ❖ 15 nm: lower dichroic signal

PREPARED SAMPLES

Fe thickness (nm)	Hole	XMCD after EMCD
10	No	In progress
10	Yes	In progress
15	Yes	Yes
15*	No*	Yes*

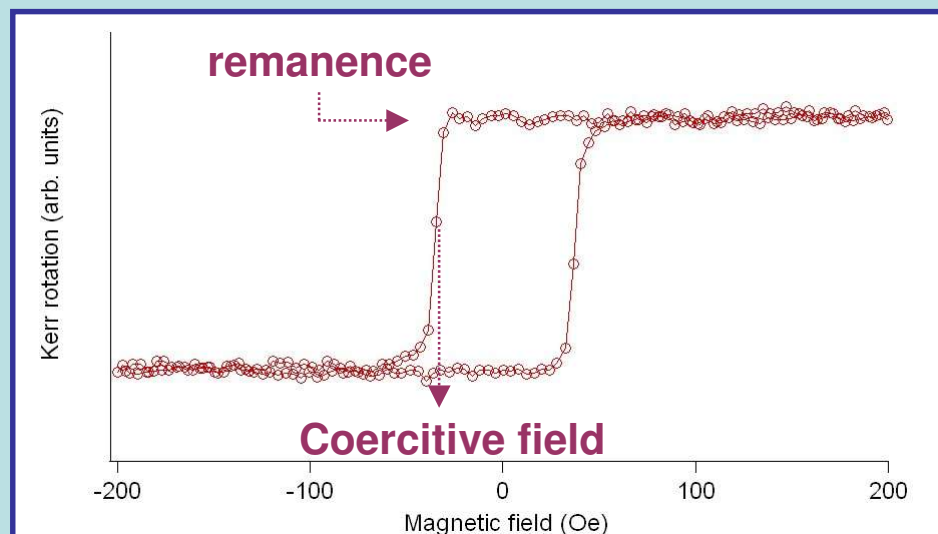
**Sample with no hole was thinned again in Wien because Cu capping was too thick*

MOKE ANALYSIS



Rotation of polarization plane of reflected light is related to magnetization

Measurement of hysteresis loops



100% remanence



Easy axis along $[100]$

A LITTLE GLANCE AT XMCD TECHNIQUE



J. Stöhr et al., *New Direction in Research with 3rd generation X-ray Synchrotron Radiation Source*, 221-250, Kluwer Academic Publishers (1994)

1^o step

Absorption of X-ray

2p core shells → 3d empty states



2^o step

Spin-orbit coupling on 2p levels

Polarized X-Rays excite spin polarized e⁻ with different rate

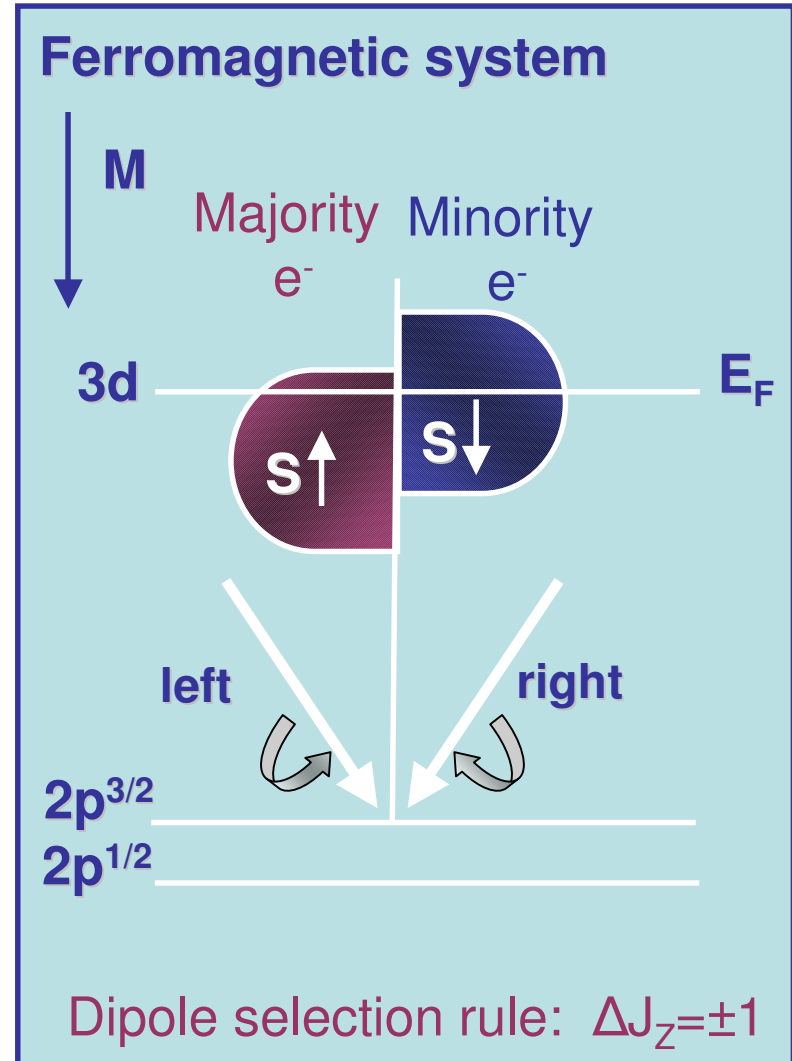
core shells ↔ source

3^o step

Exchange interaction on 3d band

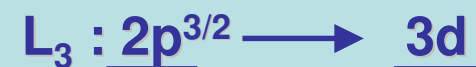
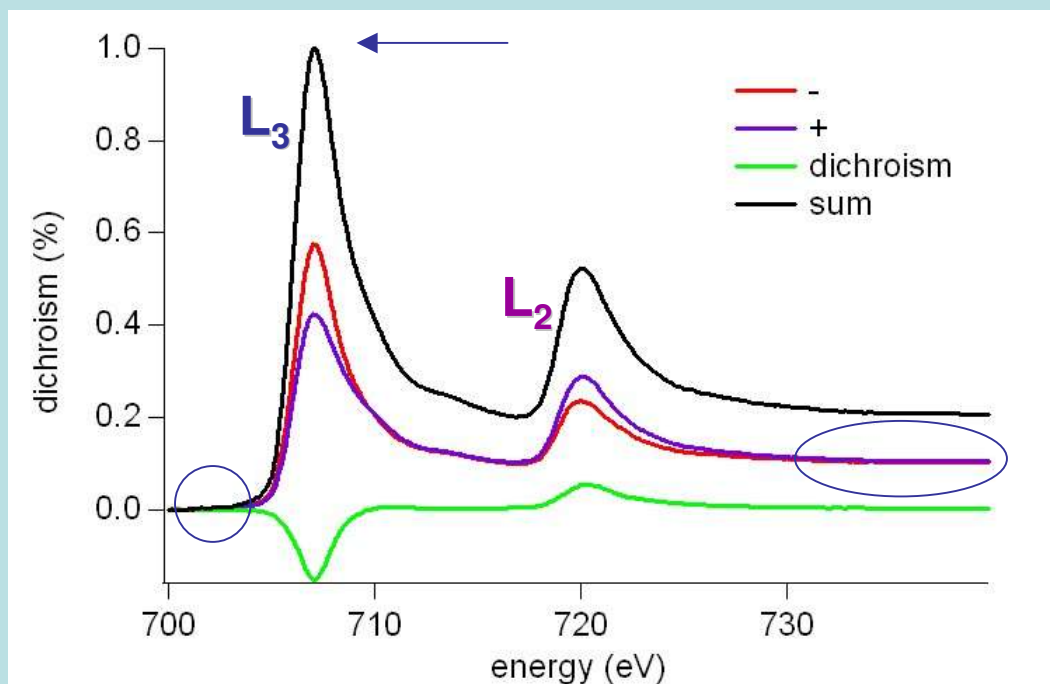
Absorption of one spin polarization is favored

Empty levels ↔ filter



XMCD STEP BY STEP

- ❖ Background removal
- ❖ Normalize post edge of + and - spectra to the same value
- ❖ Normalize sum spectrum maximum to unity
- ❖ Calculate difference spectrum $I^+(E) - I^-(E)$
- ❖ Rescale difference for polarization and sample orientation



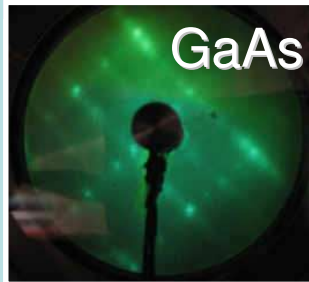
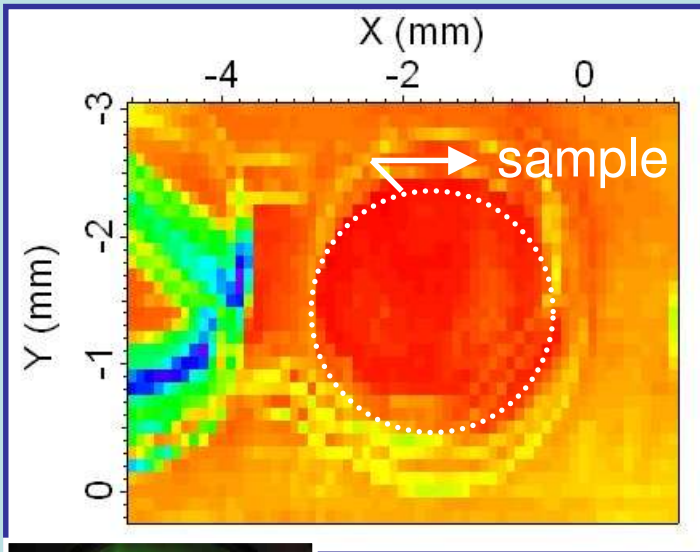
$$\text{Dichr}(\%) = \frac{I^+(E) - I^-(E)}{I^-(E) + I^+(E)}$$

XMCD EXPERIMENTS ON 15nm Fe FILMS



Before EMCD

No hole

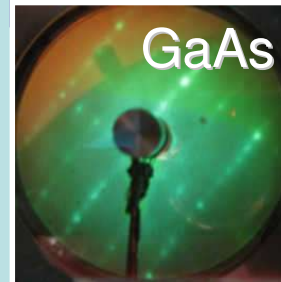
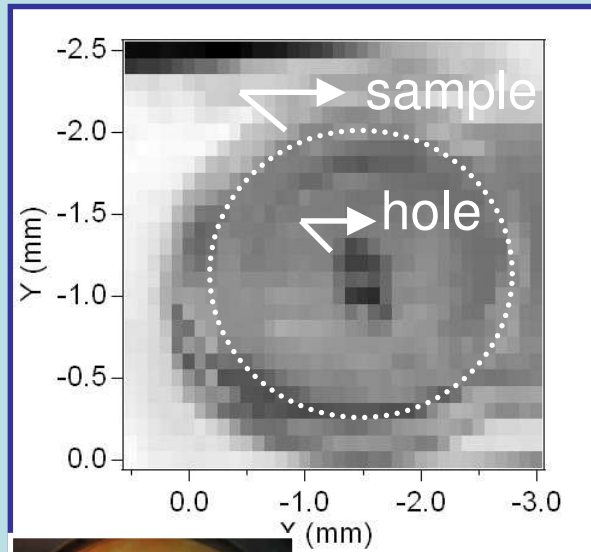


GaAs

$[110]$ $[1\bar{1}0]$

- ❖ Lower Fe signal
- ❖ % dichroism 12÷16%

Hole



GaAs

$[110]$
 $[1\bar{1}0]$

Better surface quality

- ❖ Higher Fe signal
- ❖ % dichroism 27÷33%

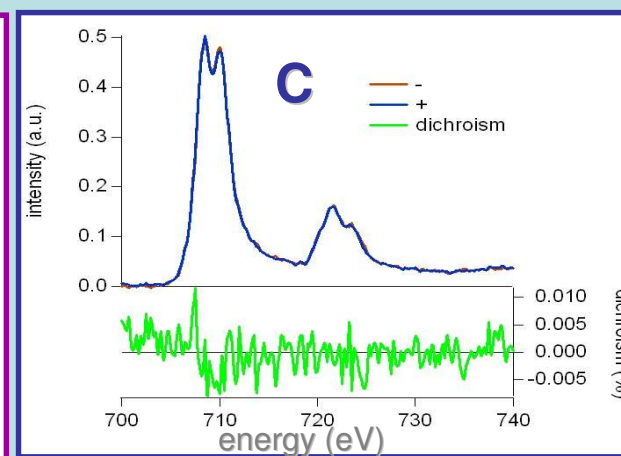
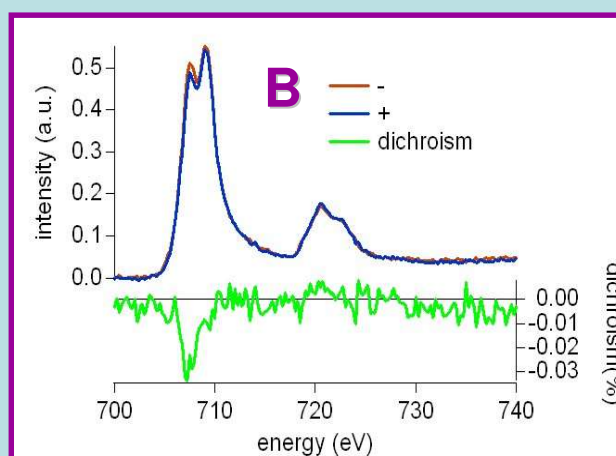
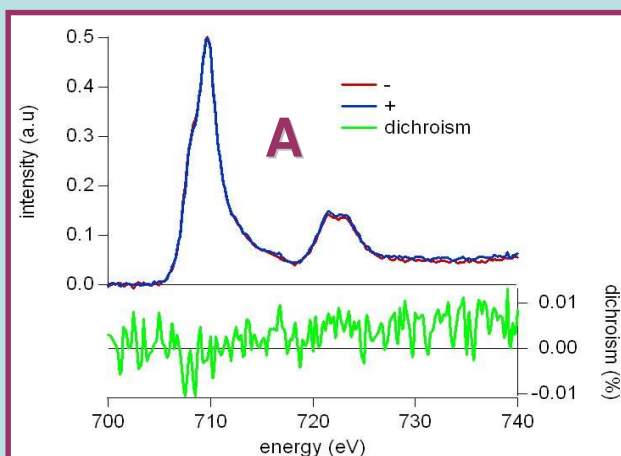
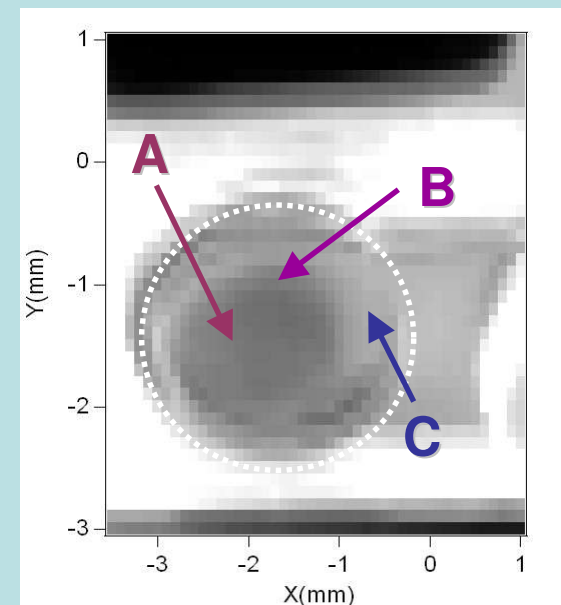
No hole

❖ Dichroism :

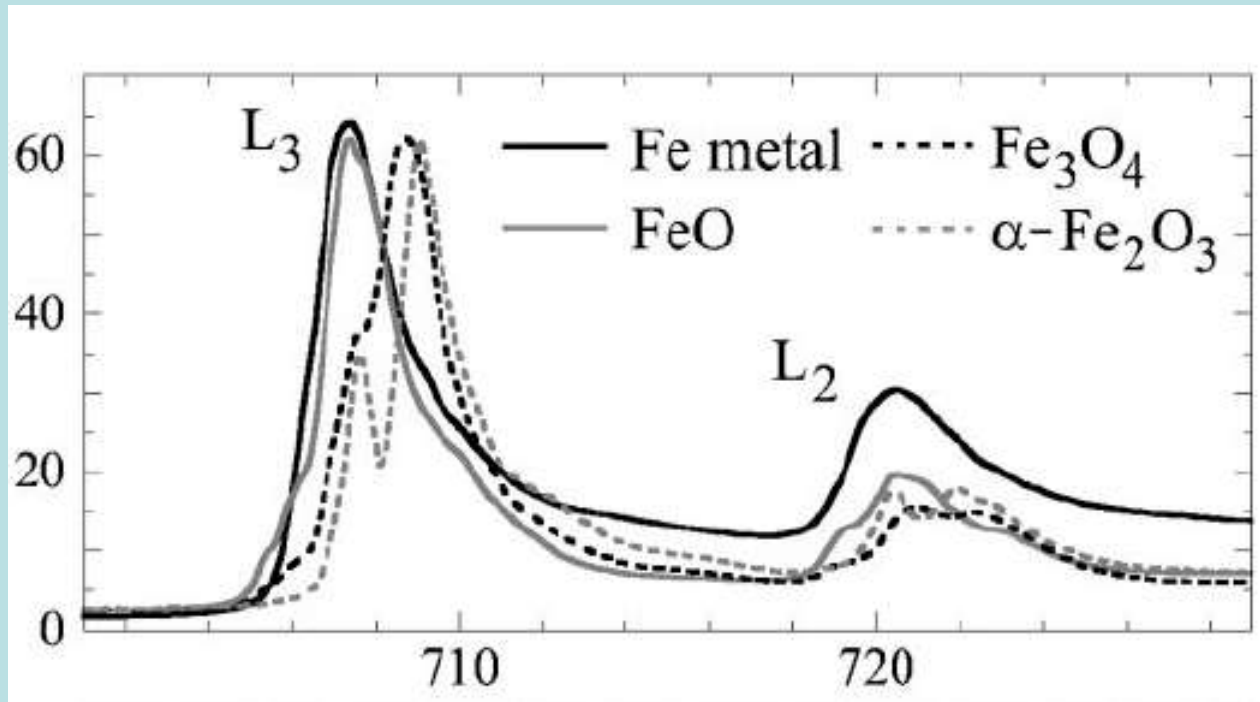
Not uniform over the surface and smaller (~3%)

❖ Preliminary analysis :

mixture of different iron oxides (Fe_3O_4 ; Fe_2O_3 ; FeO).

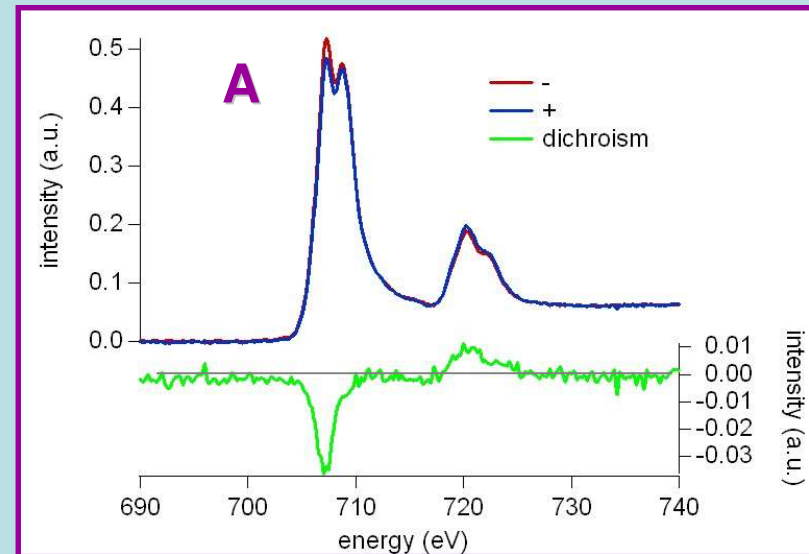
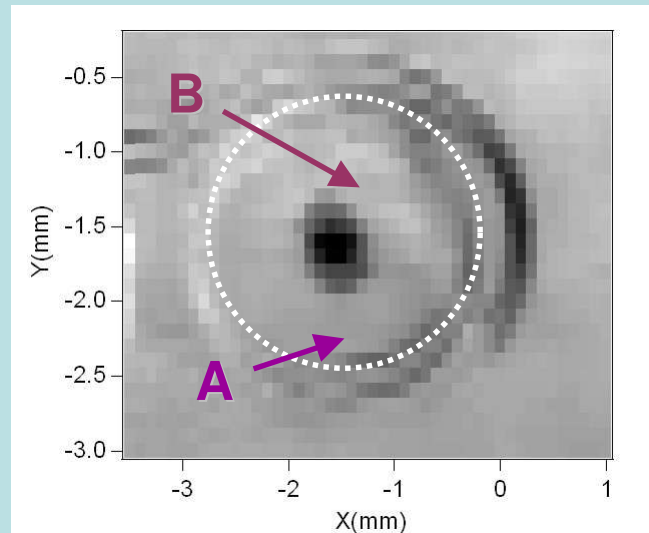


Iron oxides L_3 and L_2



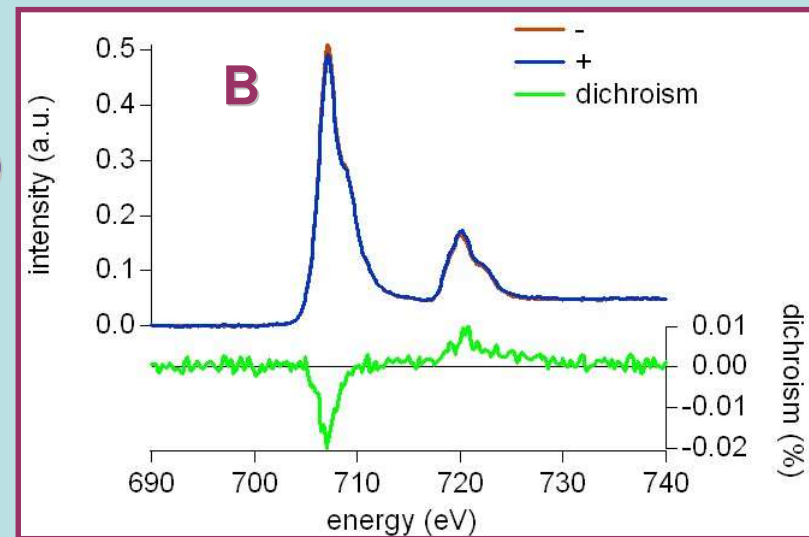
Regan et al., Phys. Rev. B, 64, 214422 (2001)

Hole



❖ Dichroism :
uniform over the surface but smaller (~2%)

❖ Preliminary analysis :
mixture of different iron oxides (Fe_3O_4 ;
 Fe_2O_3 ; FeO) and metallic Fe.



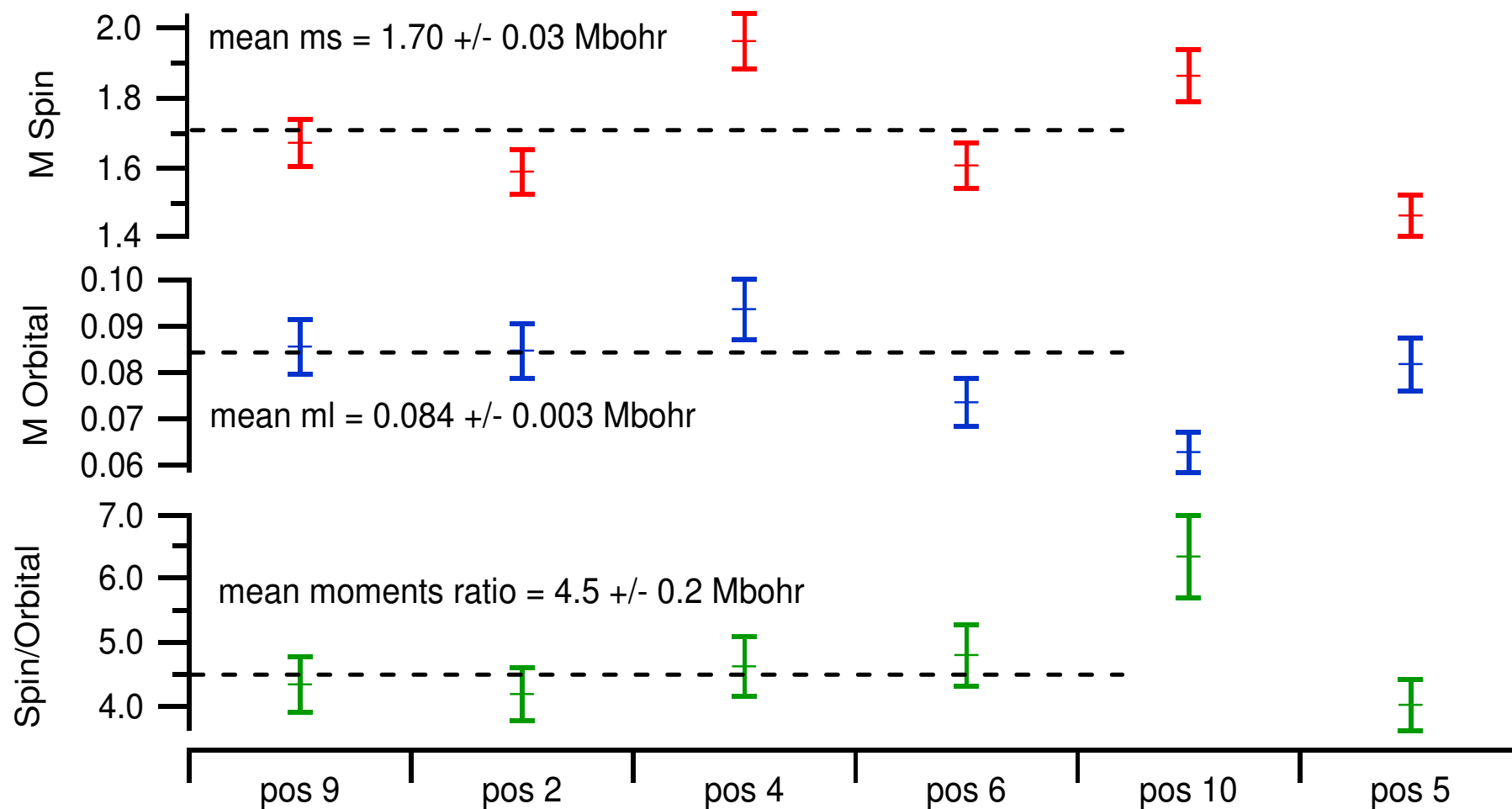
CONCLUSIONS

- ❖ Thin Fe films have been prepared with different thickness to compare the theoretical expectation of EMCD dependence on TEM specimen thickness.
- ❖ All Fe films show good crystalline quality
- ❖ XMCD experiments before and after EMCD have been performed to study ion beam milling influence and to test the different probing depth.

FUTURE

- ❖ Improvement of capping procedures
- ❖ XAS/XMCD microscopy with Fresnel diffractive optic (spot ~100nm)
- ❖ Quantitative comparison between XMCD and EMCD
- ❖ Study of EMCD as a function of applied magnetic fields.
- ❖ New systems

Calculations of magnetic moments



Total M=1.70+0.084= 1.784 Mbohr