

# Neutron Scattering of Nanostructured Magnets

Ivan K. Schuller



**DEPARTMENT OF ENERGY**

# *NEUTRON SCATTERING*

## Advantages

- Old Technique
- Interpretation Straight Forward
- Deep Penetration

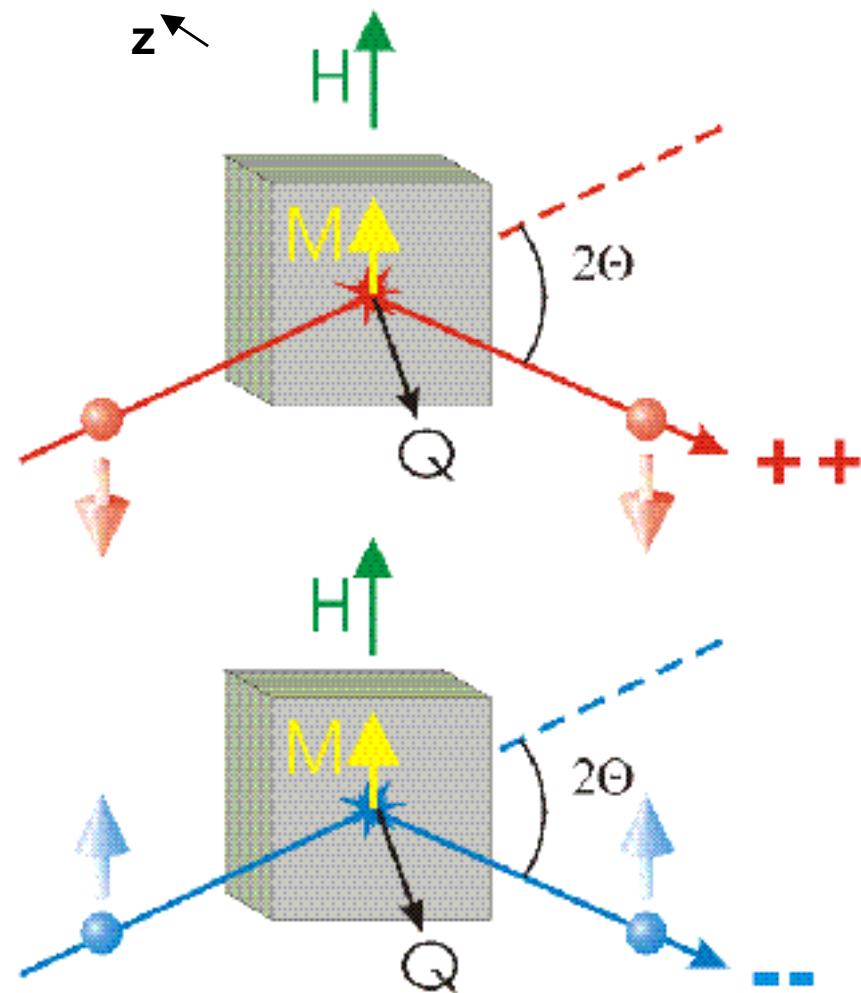
## Disadvantages

- Low Intensity
- Politics

Neutron Scattering  
Friends and phones



# Polarized Neutron Reflectometry



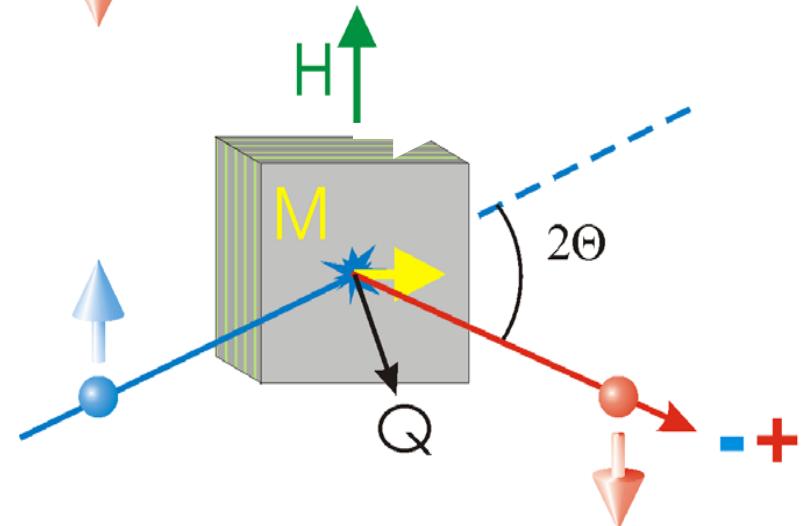
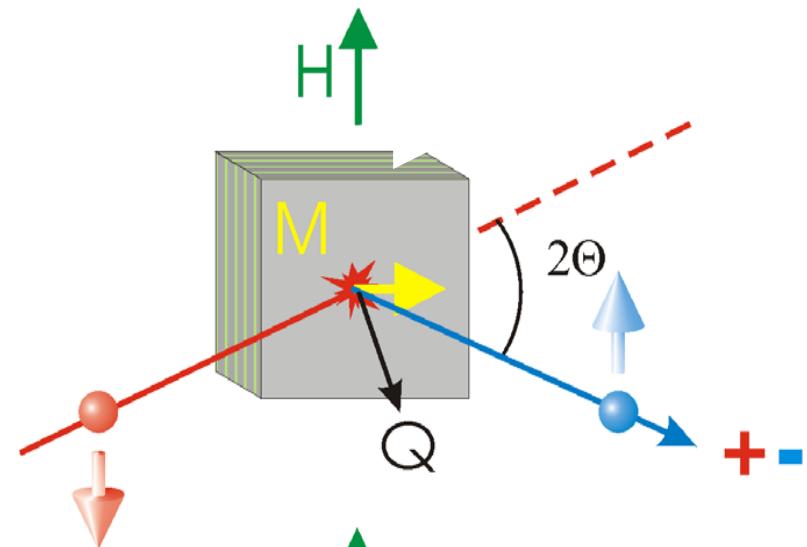
**Non spin-flip cross-sections**

- **M||H**
- **vs. Q (vs z).**

# Polarized Neutron Reflectometry

## Spin-flip cross-sections

- $M \perp H$
- vs  $Q$  (vs  $z$ )



# 3 cases

**1.- Burried Interfaces**

**(Magnetic and Nonmagnetic)**

**Roughness in Superlattices**

**2.- Nanoscopic Magnetic Ordering**

**Magnetic Orientation in Multilayers**

**3.- Vector Magnetometry**

**Exchange Bias in Bilayers**

# Magnetic & Nonmagnetic Roughness

## Mo/Ni Superlattices

# **Collaborators**

**ORNL**

**J. Cable**

**ANL**

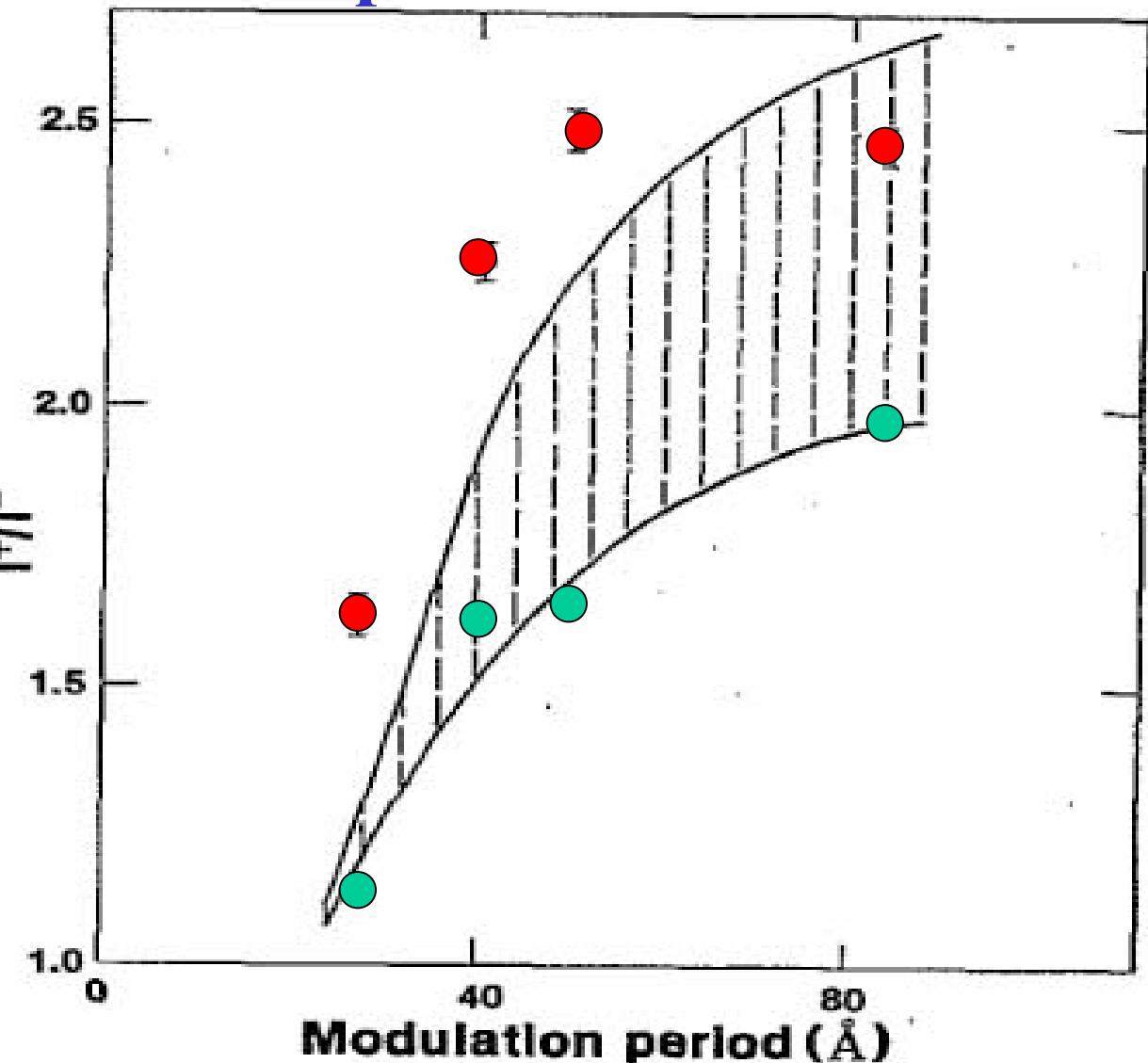
**M. Khan**

**G. Felcher**

# Polarized Neutron Scattering

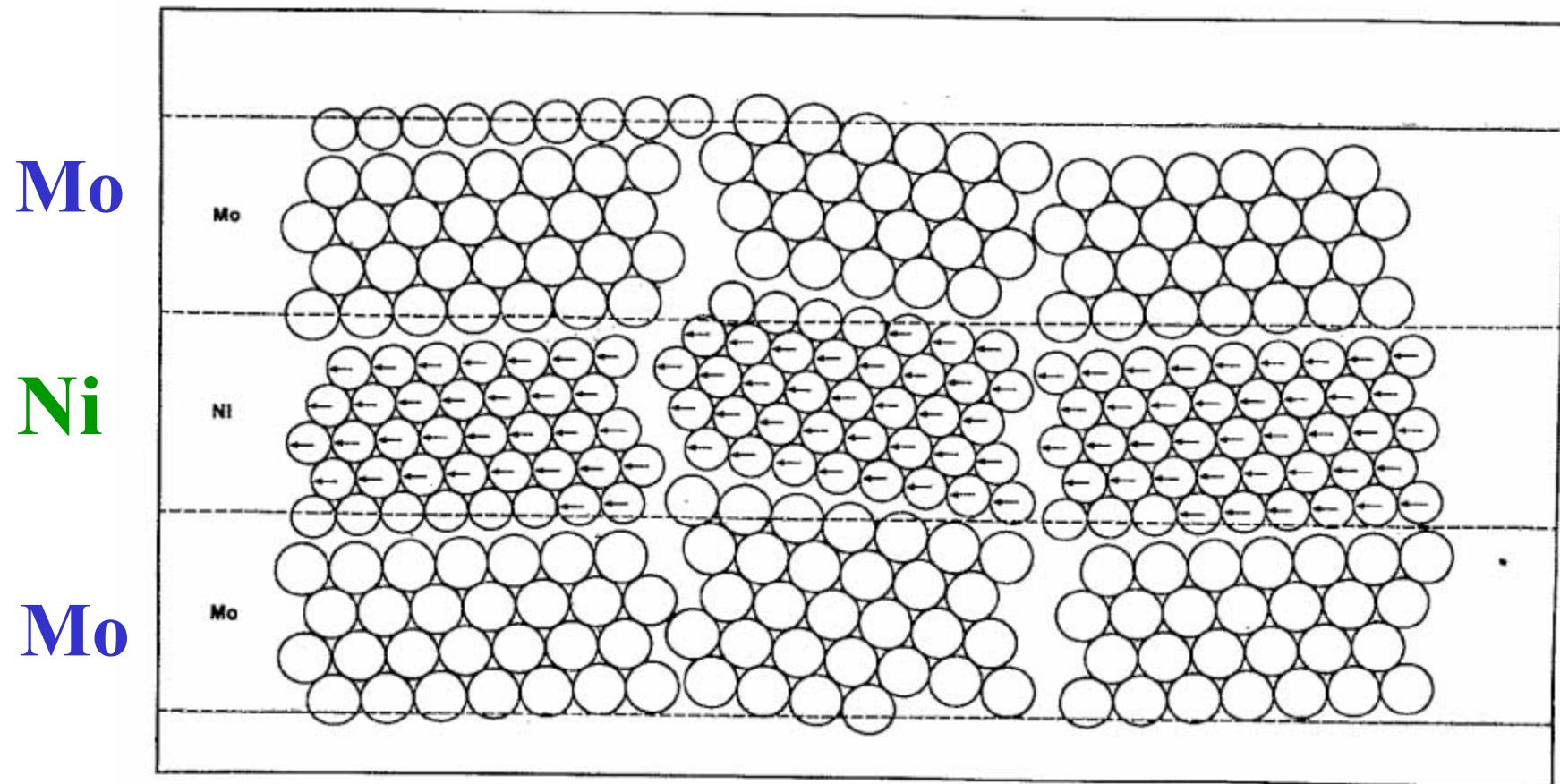
## Mo/Ni Superlattices

- Square Modulation
- All possible models
- Experimental Flipping Ratio



# Mo/Ni Superlattices

“A comparison of X-ray and neutron diffraction implies that the magnetic profile in the superlattice is more perfect than the chemical profile.”



J. W. Cable, M.R. Khan, G. Felcher, I. K. Schuller, Phys. Rev. B34, 1643(1986)

# 3 cases

1.- Burried Interfaces(Magnetic-Nonmagnetic)  
Roughness in Superlattices

2.- Nanoscopic Magnetic Ordering  
**Magnetic Orientation-Multilayers**

3.- Vector Magnetometry  
Exchange Bias in Bilayers

# Coupling

# Fe/Cr Multilayers

S. teVelthuis et al., In print 2002

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# Collaborators

**ANL**

**S. te Velthuis**

**G. Felcher**

**UCSD**

**S. Kim**

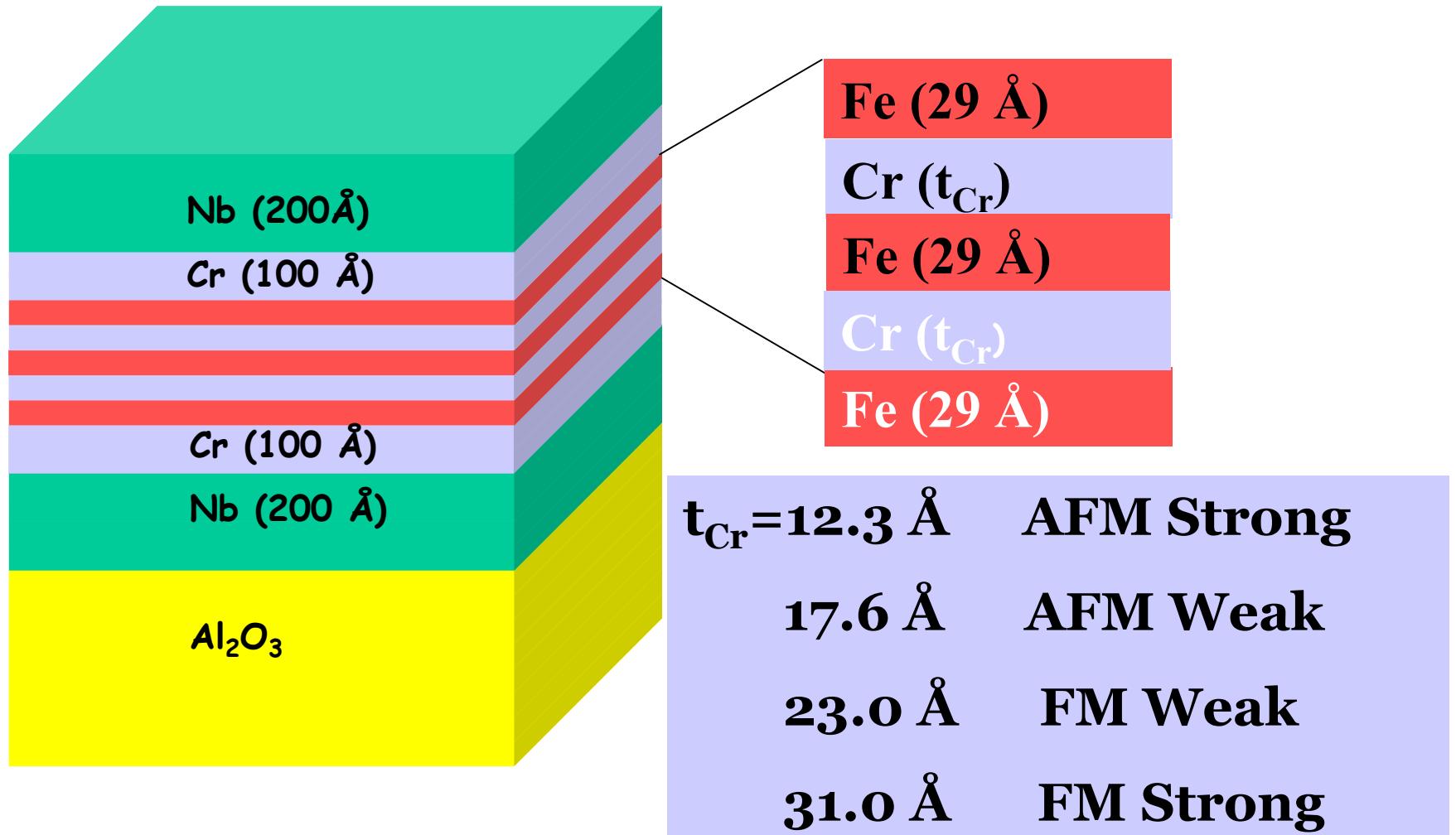
# Interlayer exchange coupling

Exchange coupling  
F layers  
across non-F spacer  
is oscillatory

- Long period      ( $\approx 18\text{\AA}$ , Fe/Cr)
- Short period      ( $\approx 2\text{\AA}$ , Fe/Cr)  
Only for very smooth interfaces

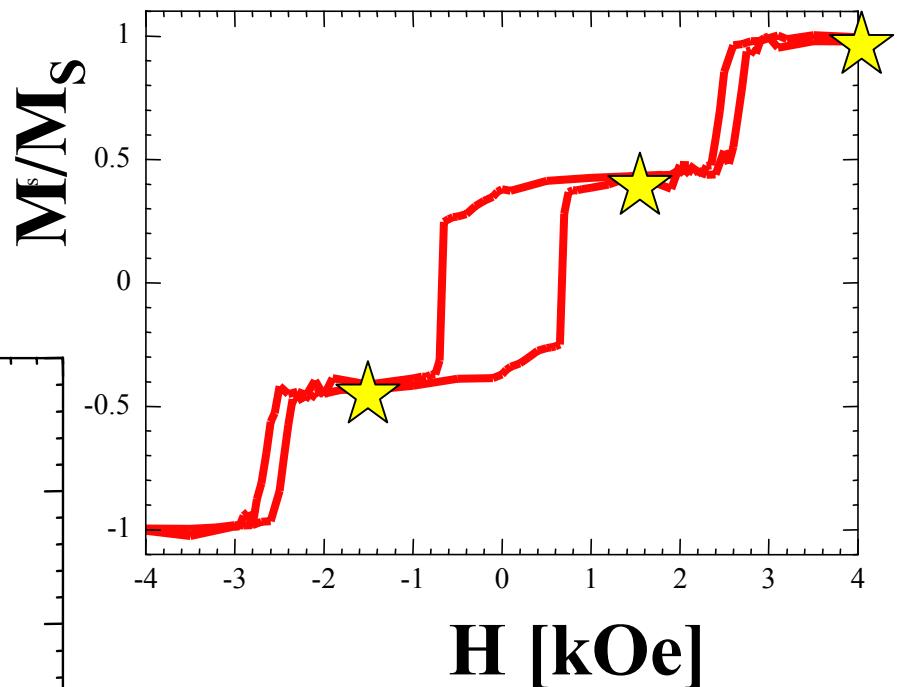
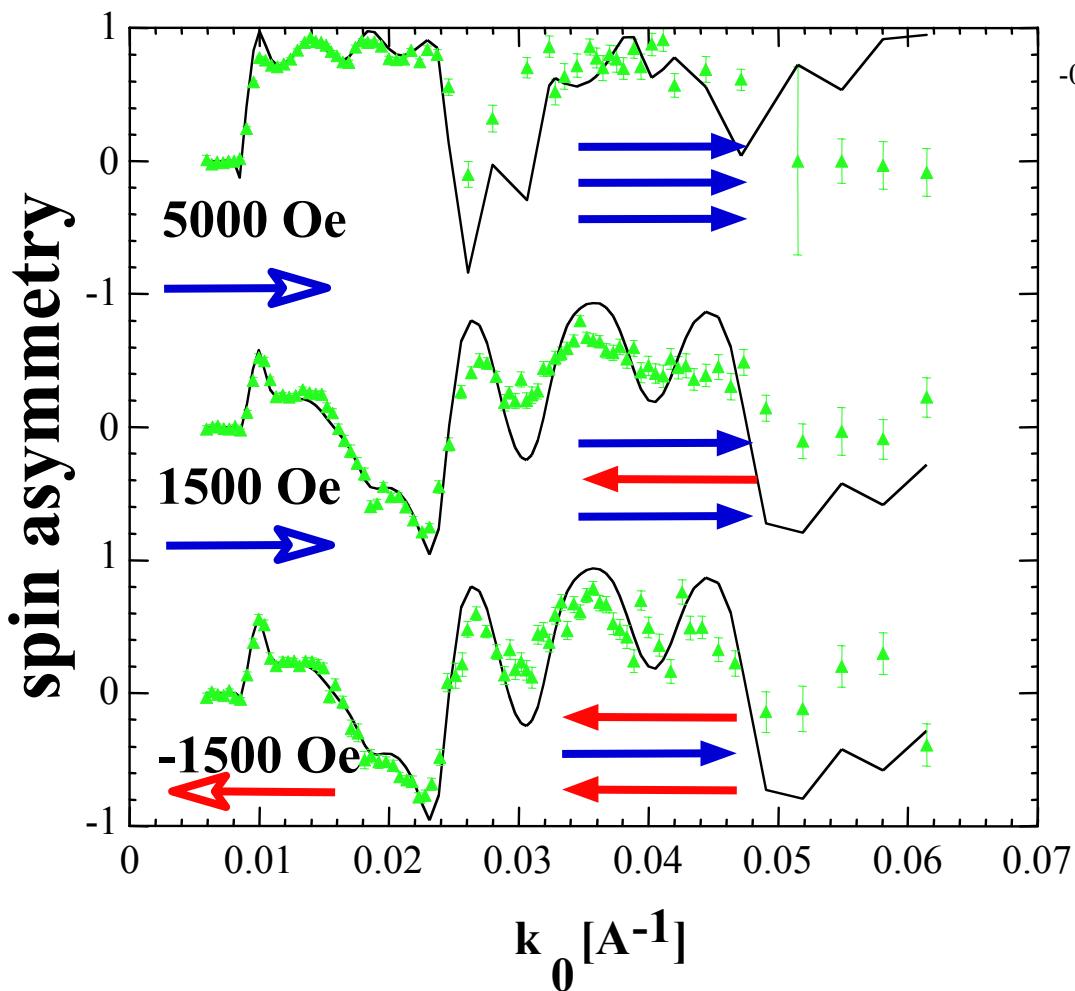
Review: M.D. Stiles JMMM 200 (1999) 322

# Epitaxial Fe/Cr (110) multilayers



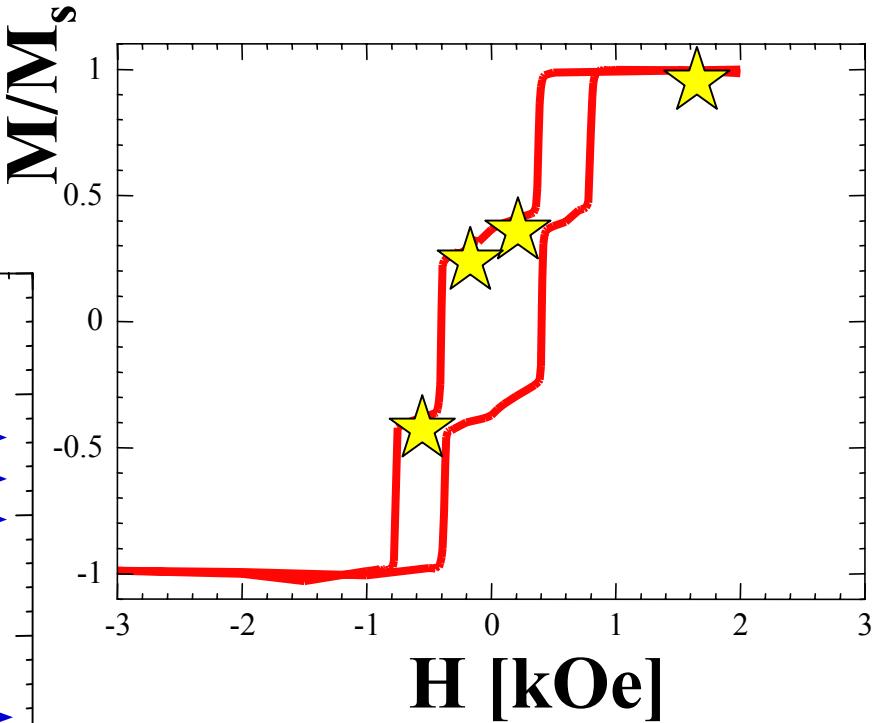
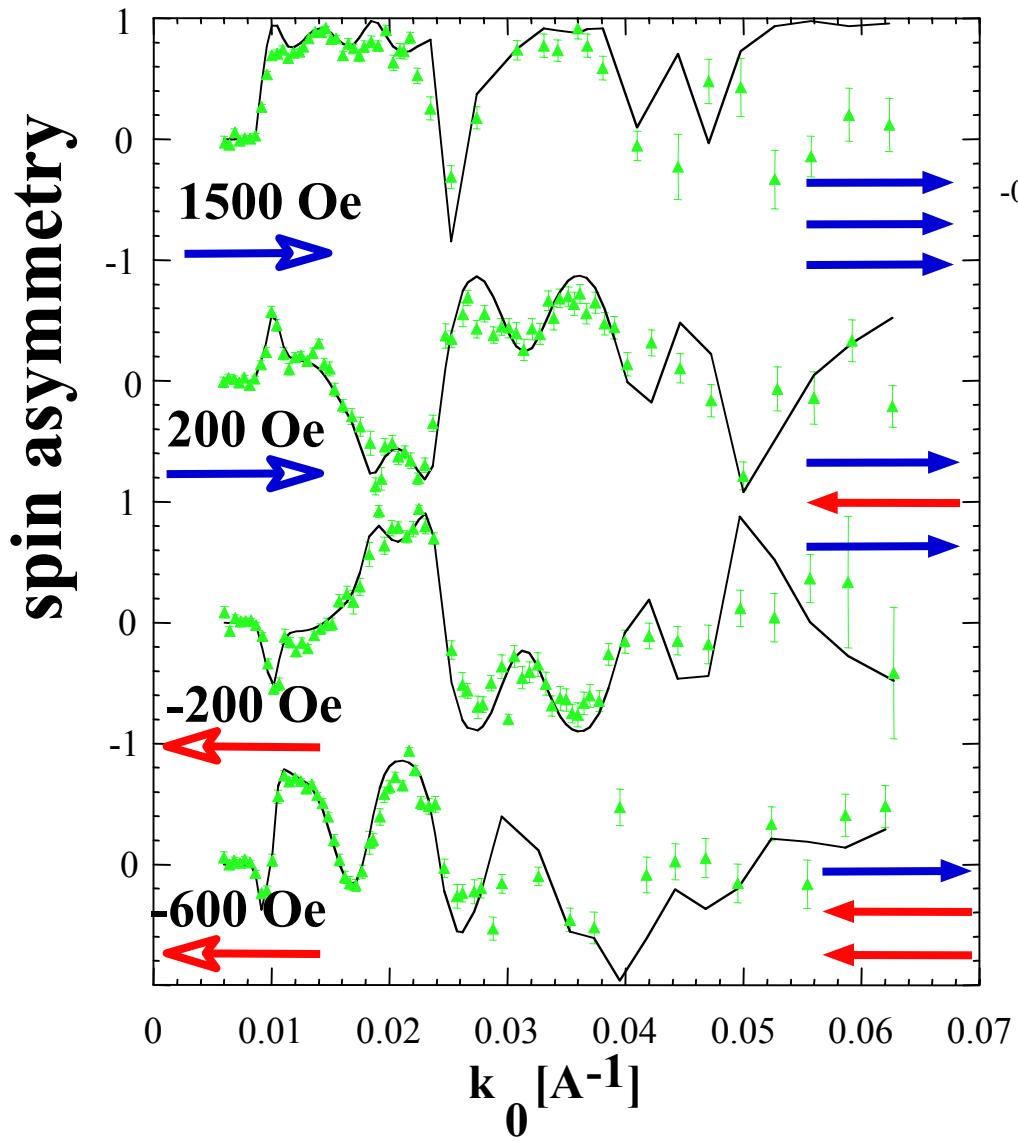
$t_{Cr} = 12.3 \text{ \AA}$

Spin asymmetry  
 $(R^+ - R^-)/(R^+ + R^-)$



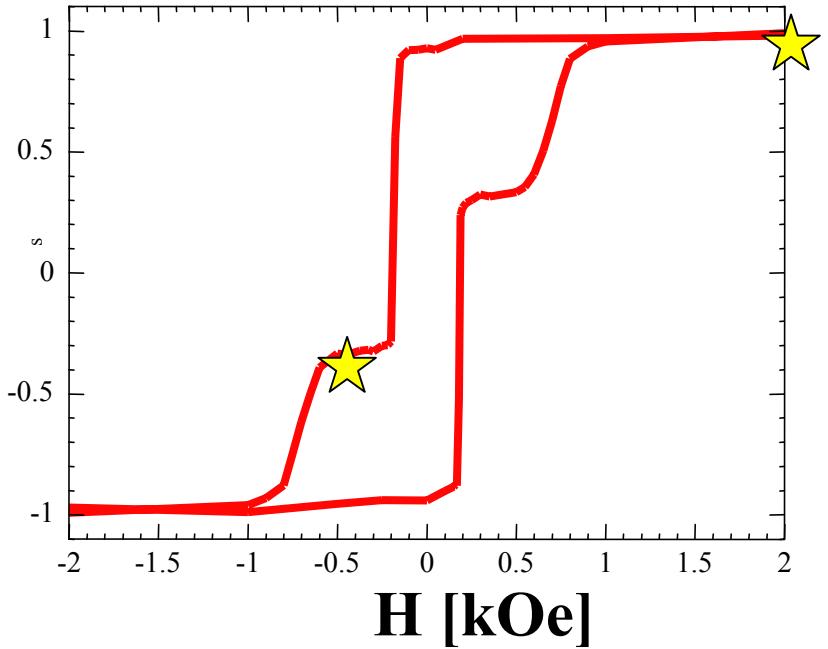
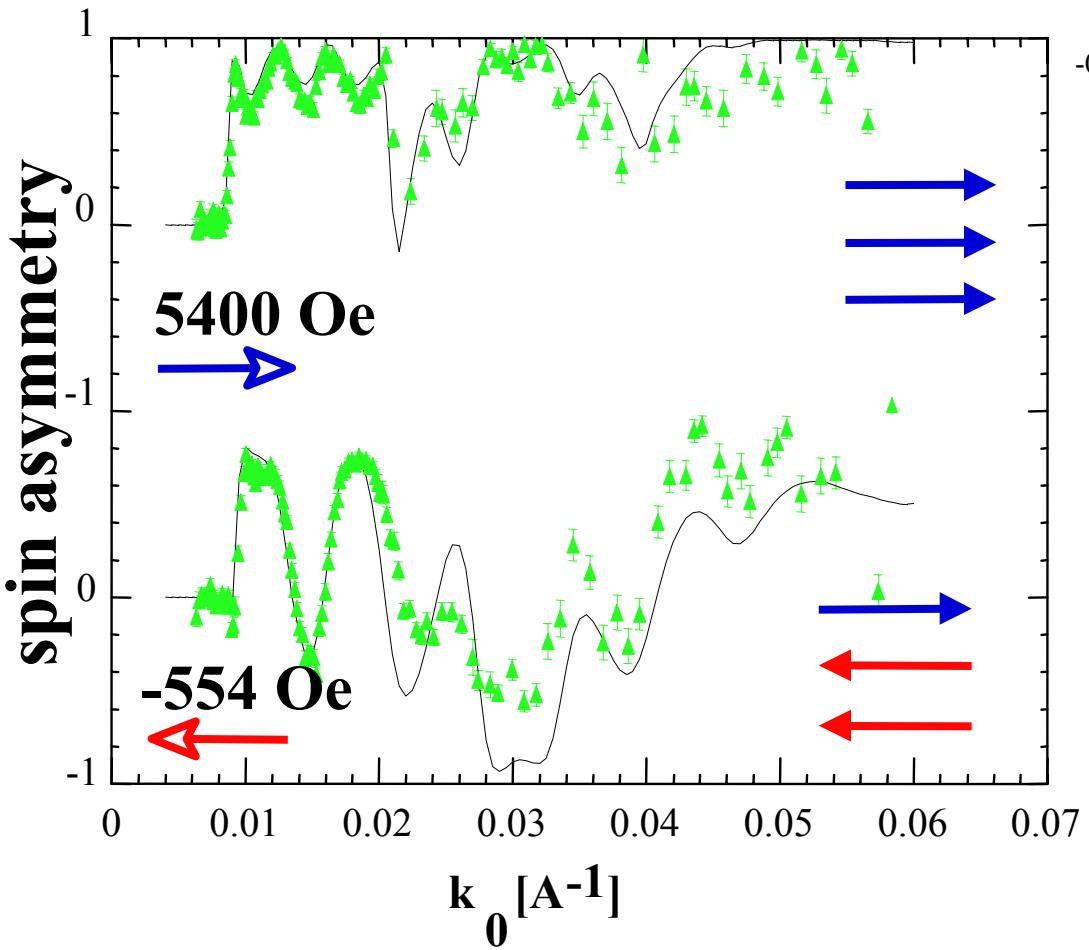
AFM Strong  
Top and Bottom  
Same

$t_{\text{Cr}} = 17.6 \text{ \AA}$

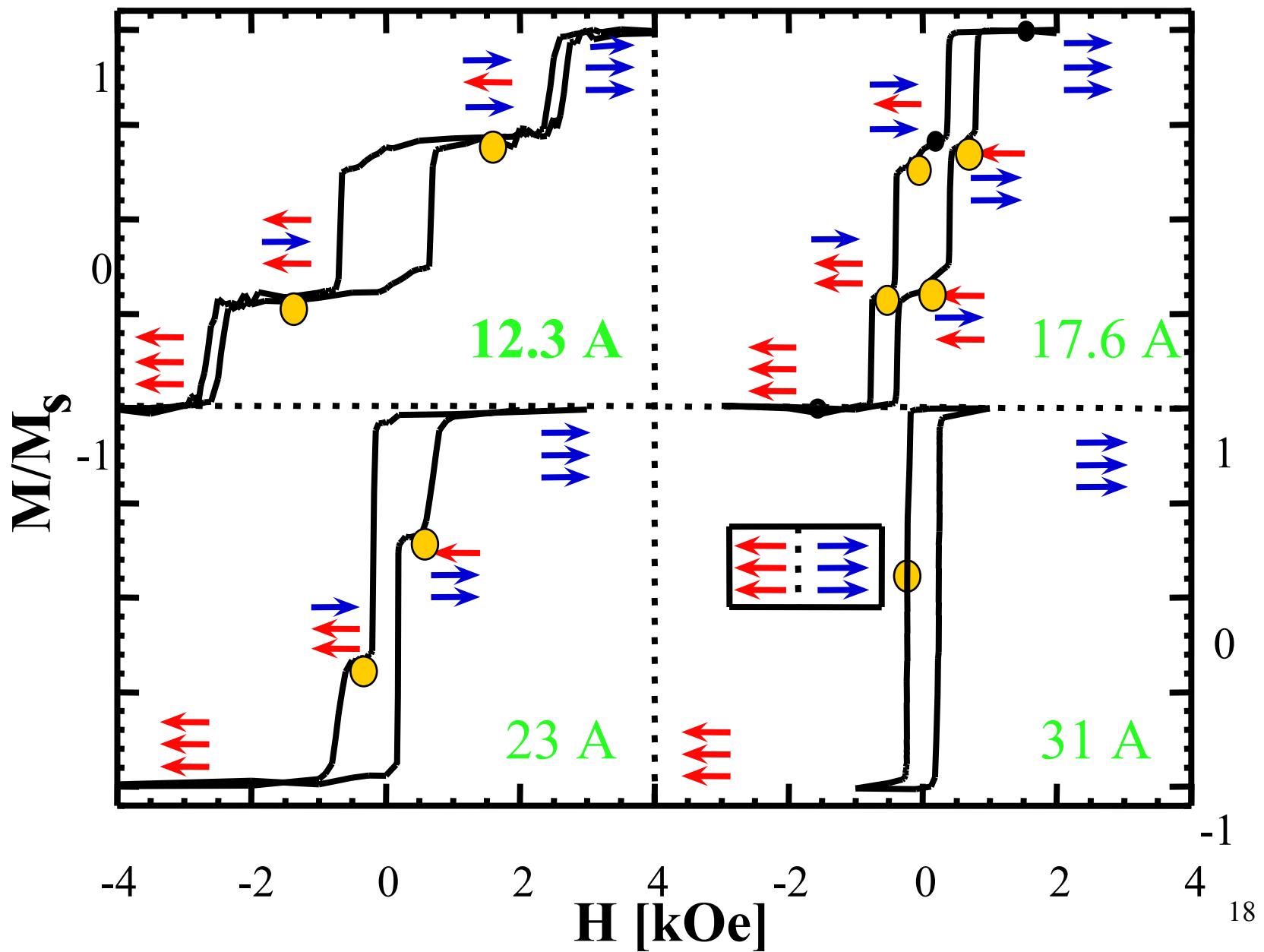


AFM Weak  
Top and bottom  
Different

$t_{\text{Cr}} = 23 \text{ \AA}$



# Summary



# 3 cases

**1.- Burried Interfaces (Magnetic and Nonmagnetic)**

**Roughness in Superlattices**

**2.- Nanoscopic Magnetic Ordering**

**Magnetic Orientation in Multilayers**

**3.- Vector Magnetometry**

**Exchange Bias in Bilayers**

# Exchange Bias

Fe- $X\text{F}_2$  Bilayers

$X=\text{Fe}, \text{Mn}, \text{Zr}, \text{Co}$

# Collaborators

**LANL:**

**M Fitzsimmons**  
**J.R. Groves**  
**P. Arendt**  
**R. Springer**  
**A. Hoffmann**  
**P.C. Yashar**

**U. A. Barcelona:**  
**J. Nogues**

**NIST:**

**C.F. Majkrzak**  
**J.A. Dura**  
**B. Shull**

**HMI:**

**H. Fritzsch**

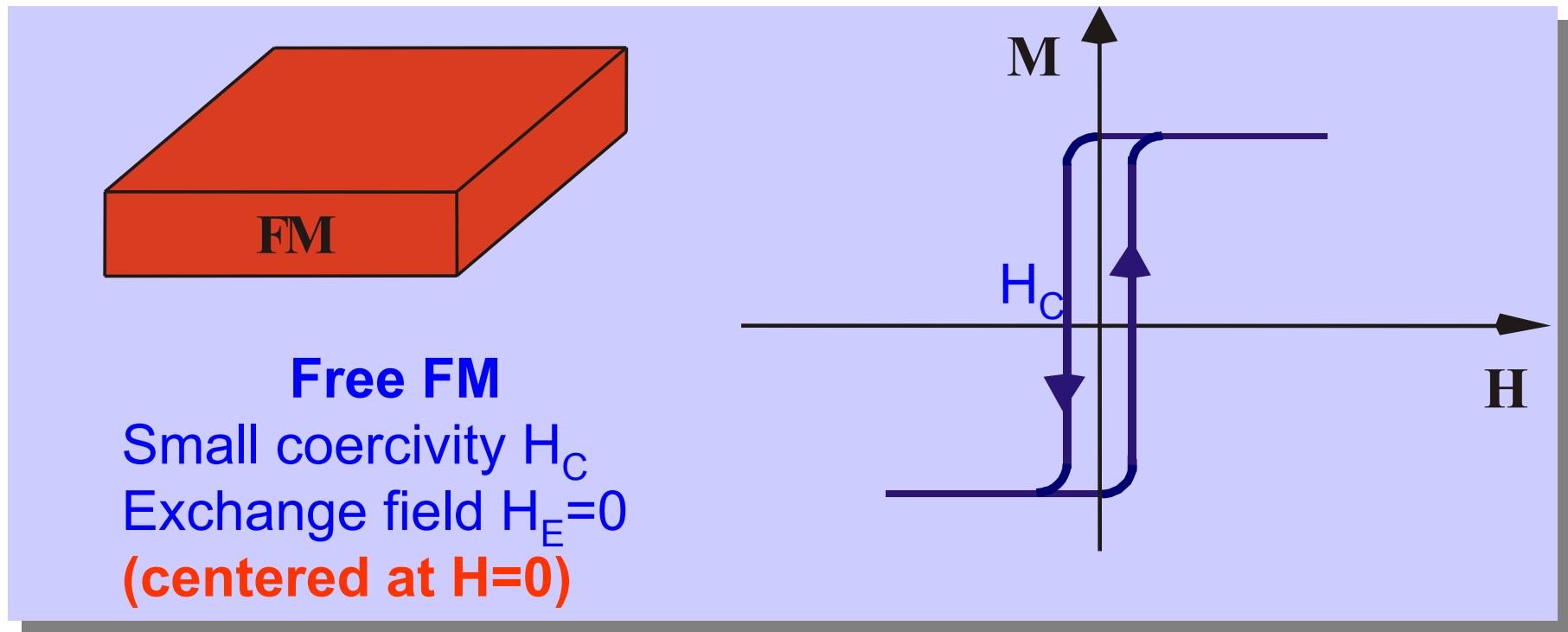
**ILL:**

**Vincent Leiner**  
**Hans Lauter**

**UCSD:**

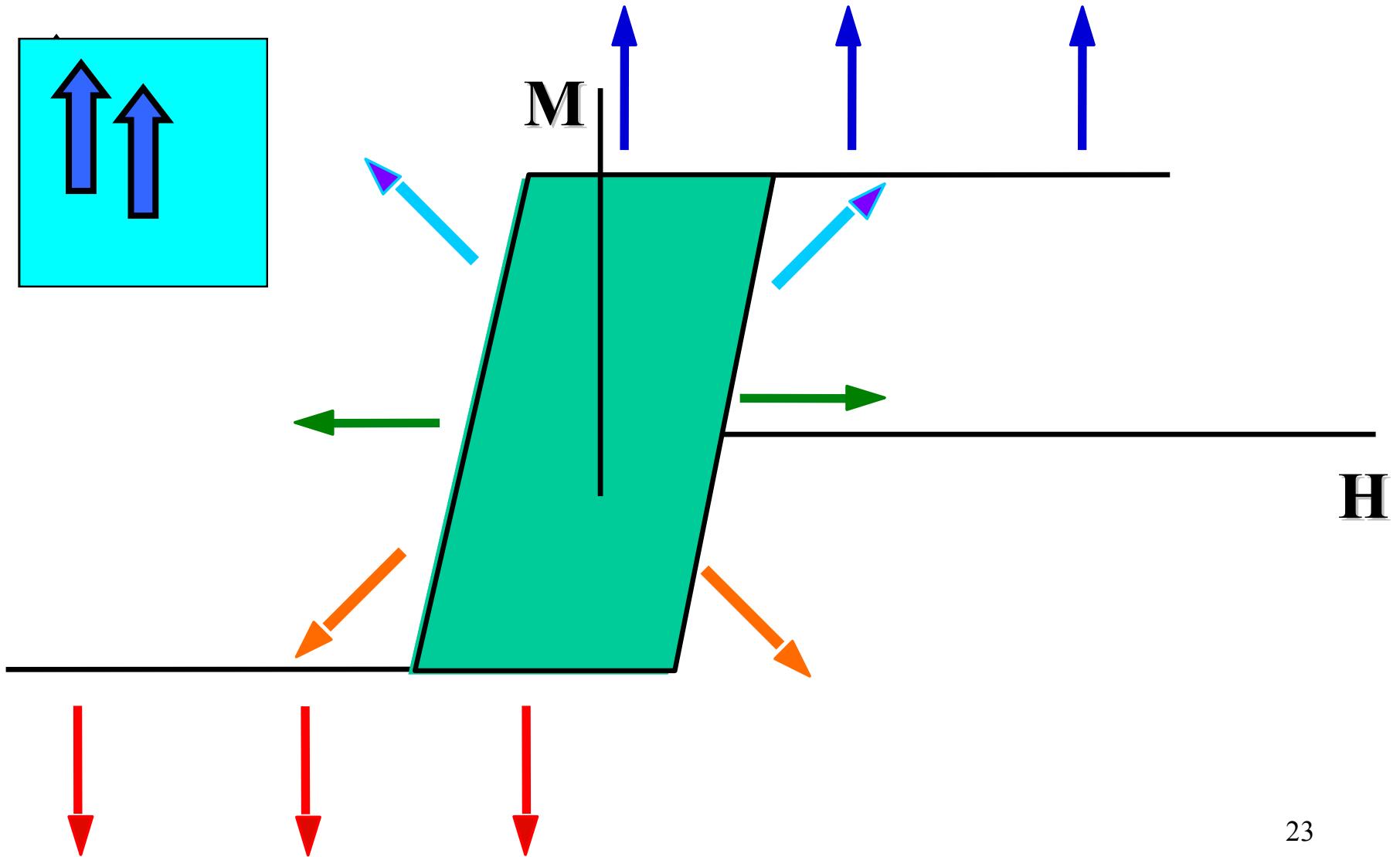
**C. Leighton**  
**K. Liu**

# EVERYTHING YOU WANTED TO KNOW ABOUT FERROMAGNETISM.....but ...were afraid to ask

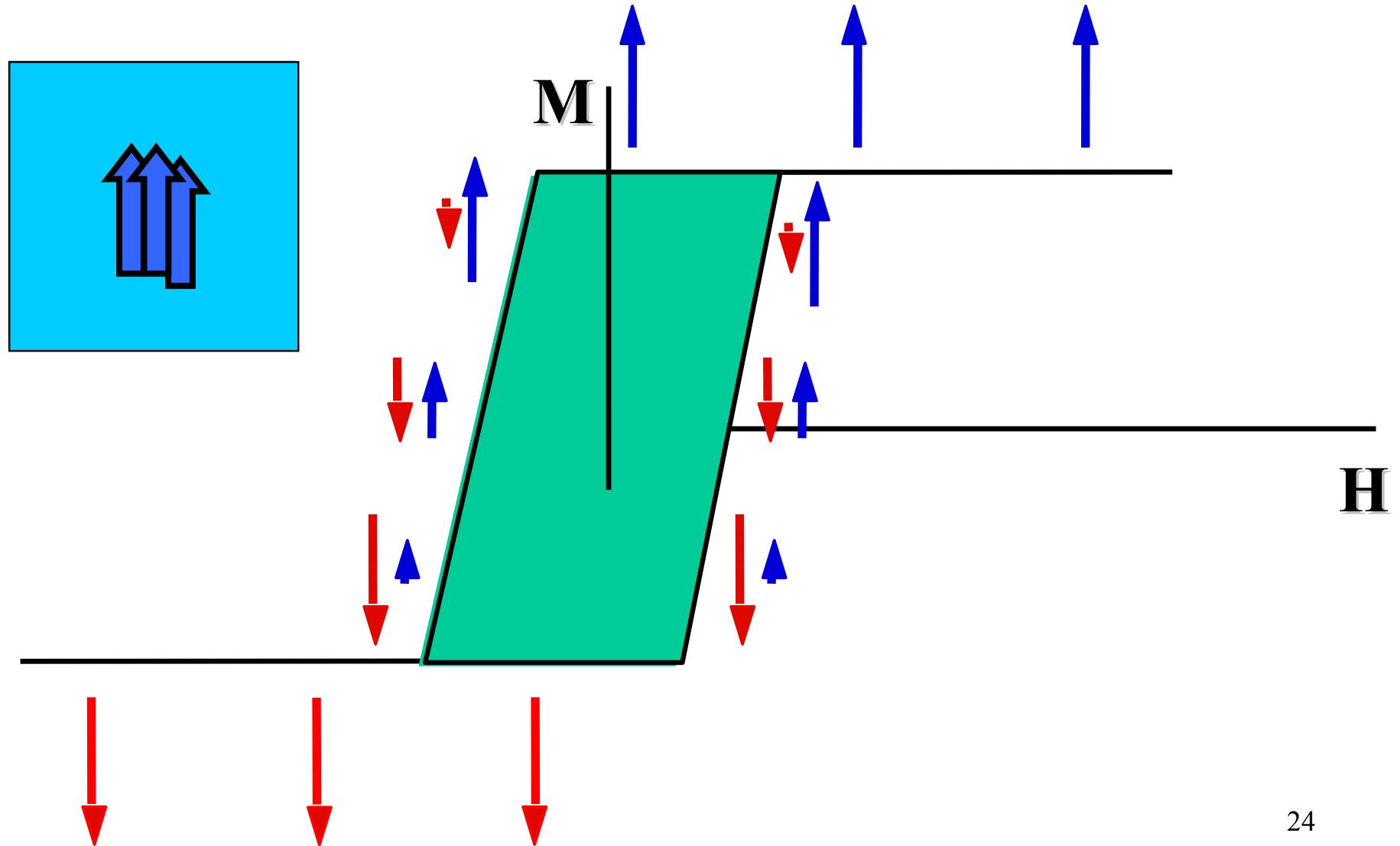


## WHAT IS THE REVERSAL MODE ?

# ROTATION



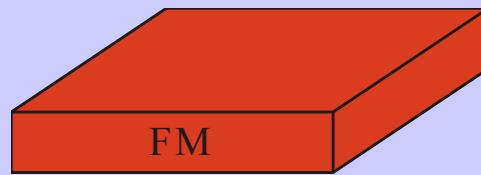
# NUCLEATION & WALL MOTION



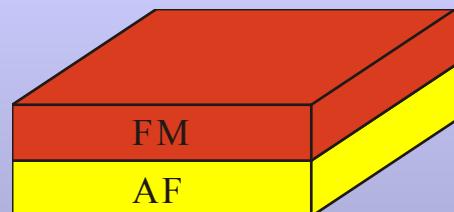
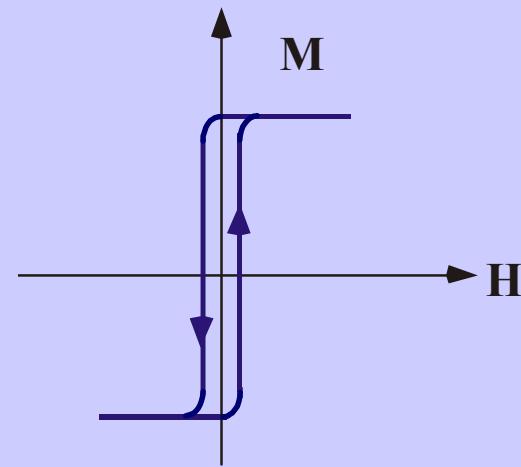
# **Reversal Mode is the Same in Both Sides of the Hysteresis Loop**

*In all materials systems  
known to man*

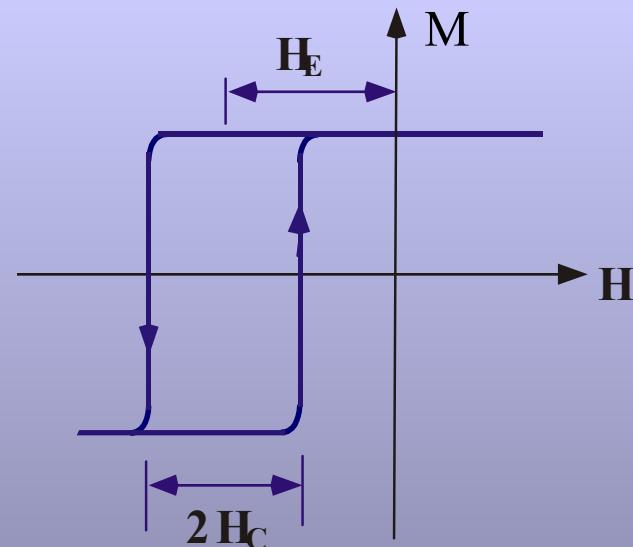
# Exchange Bias



**Free FM**  
**Exchange field  $H_E = 0$**   
**Small coercivity  $H_C$**

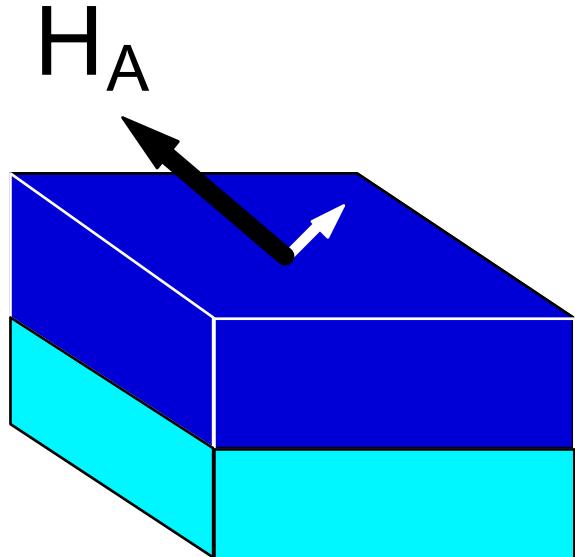


**Pinned FM**  
**Large  $H_E$**   
**Large  $H_C$**

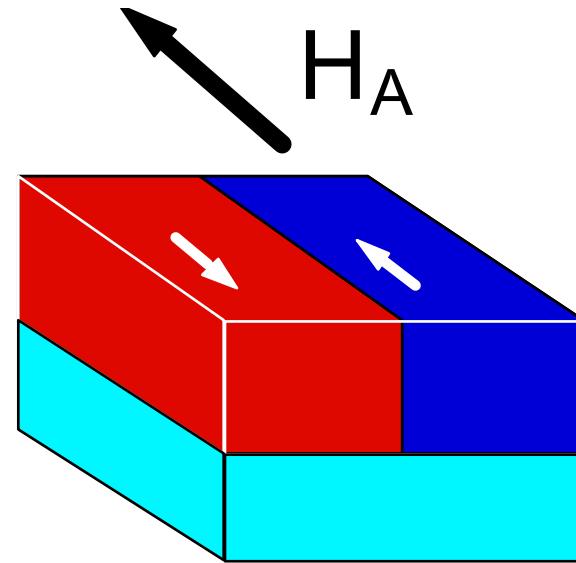


# Some Neutron Related Issues

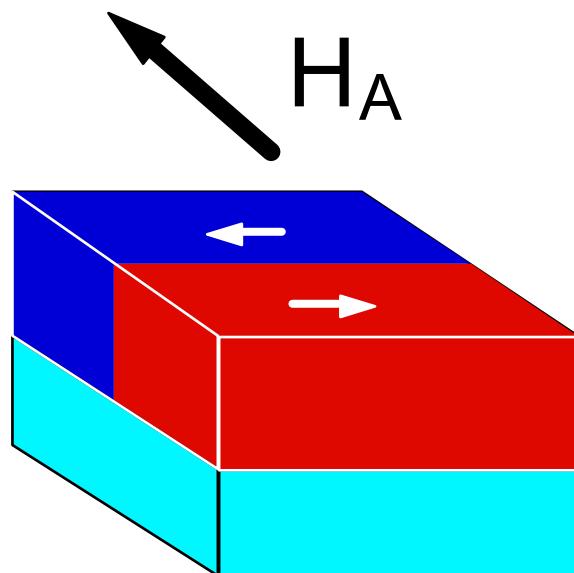
- Reversal Mode
- Domains in the F or AF
  - Moments in the AF
- Interfacial magnetic structure



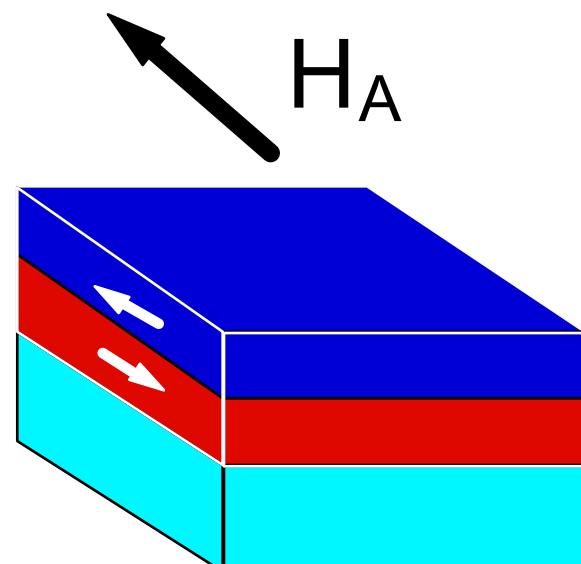
**Coherent Rotation**



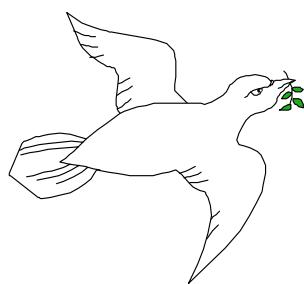
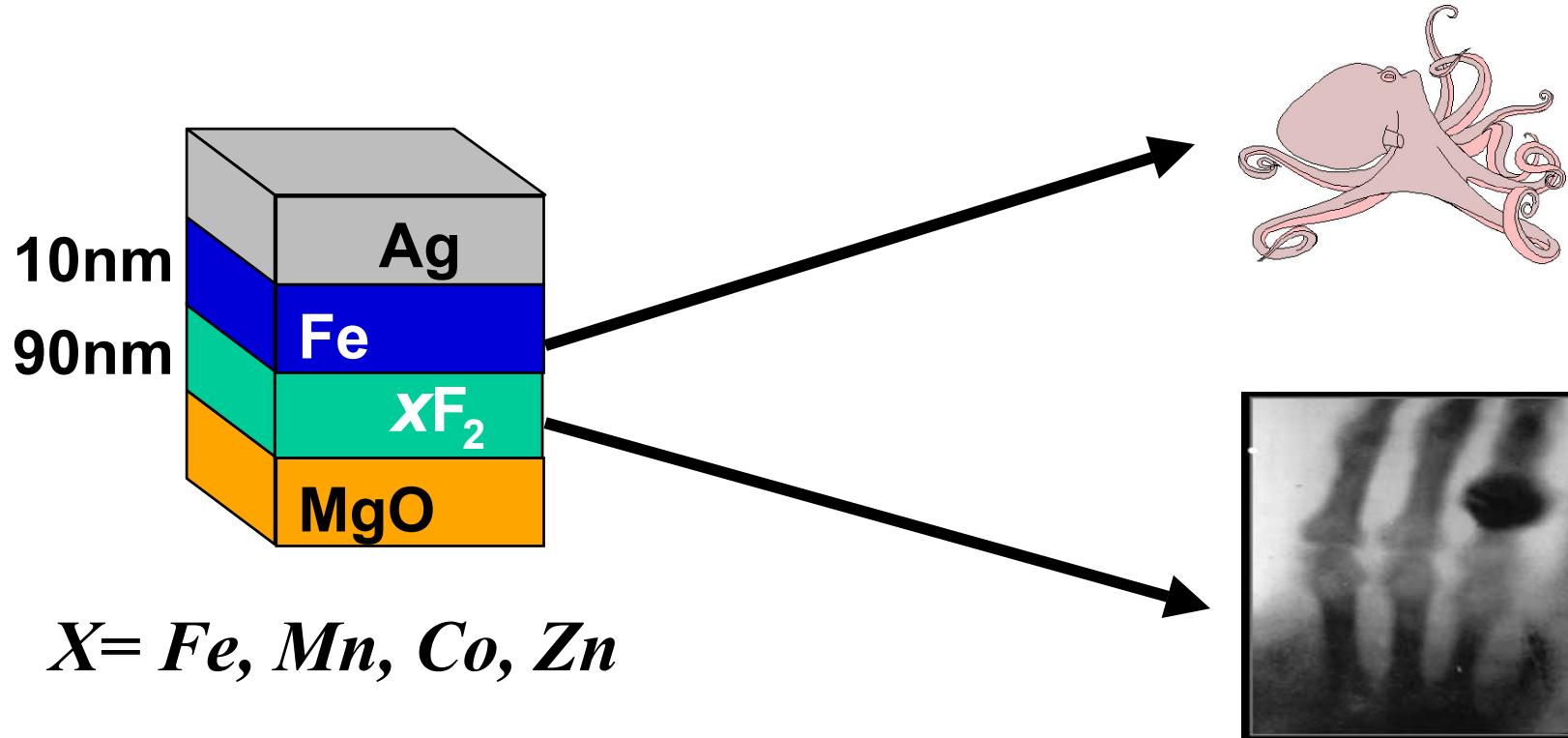
**Opposed Domains**



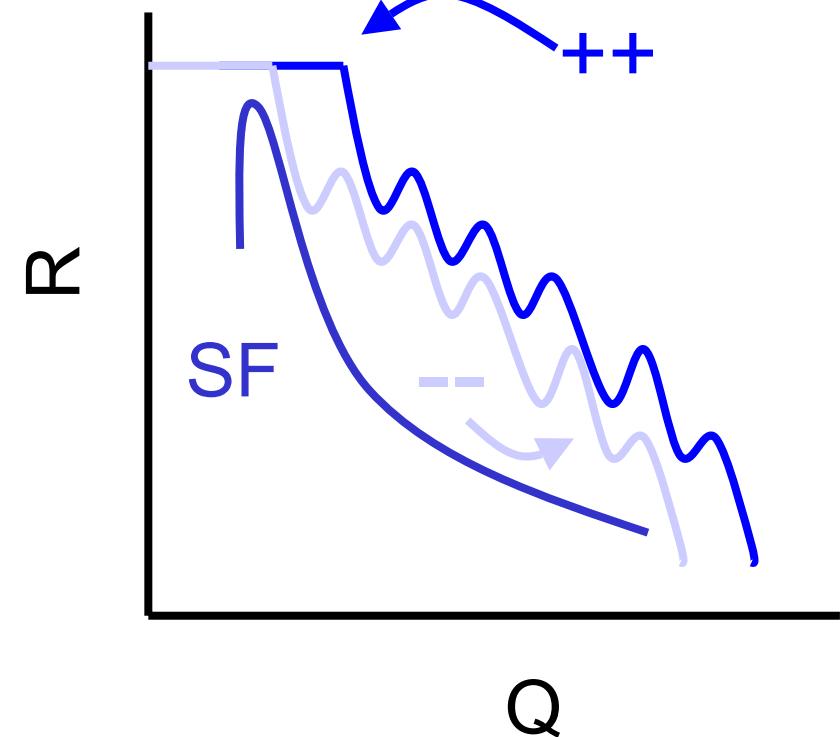
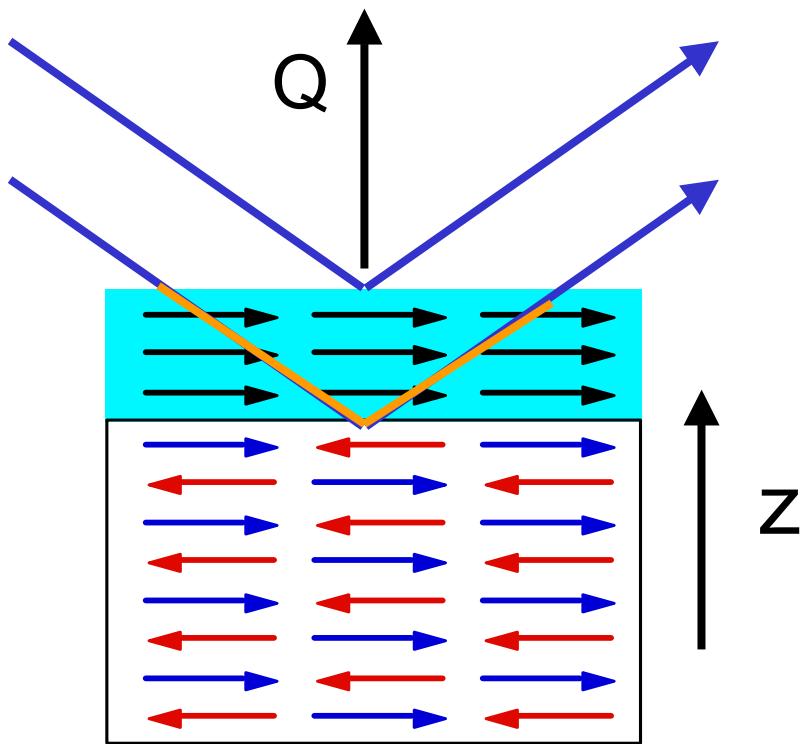
**Opposed Domains + Rotation**



**Layered Domains**

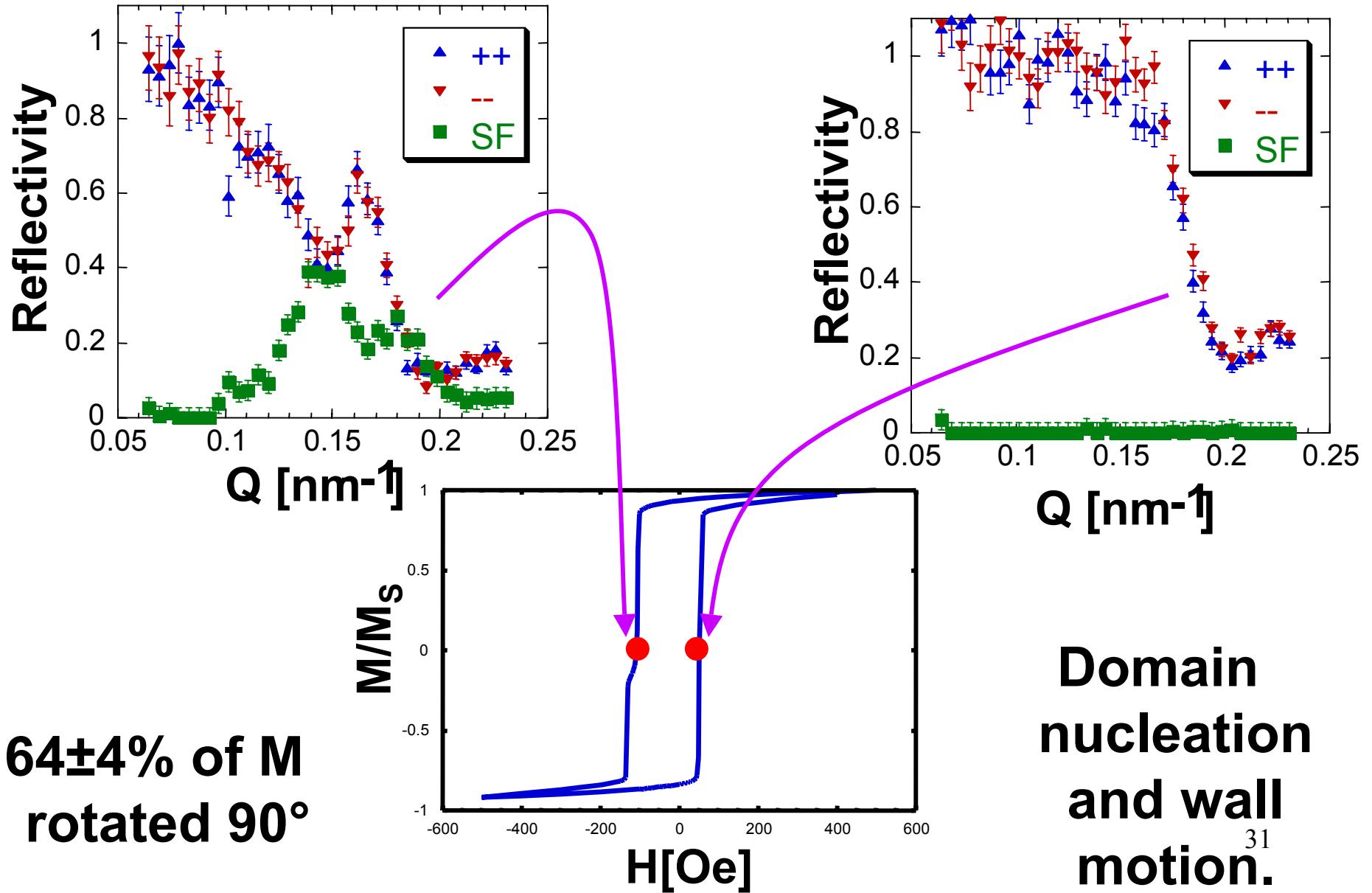


# Neutron scattering— essential!



$$\vec{M}(\vec{H}_A \quad \vec{H}_{fC}, \quad T, \quad P, \quad z \dots)$$

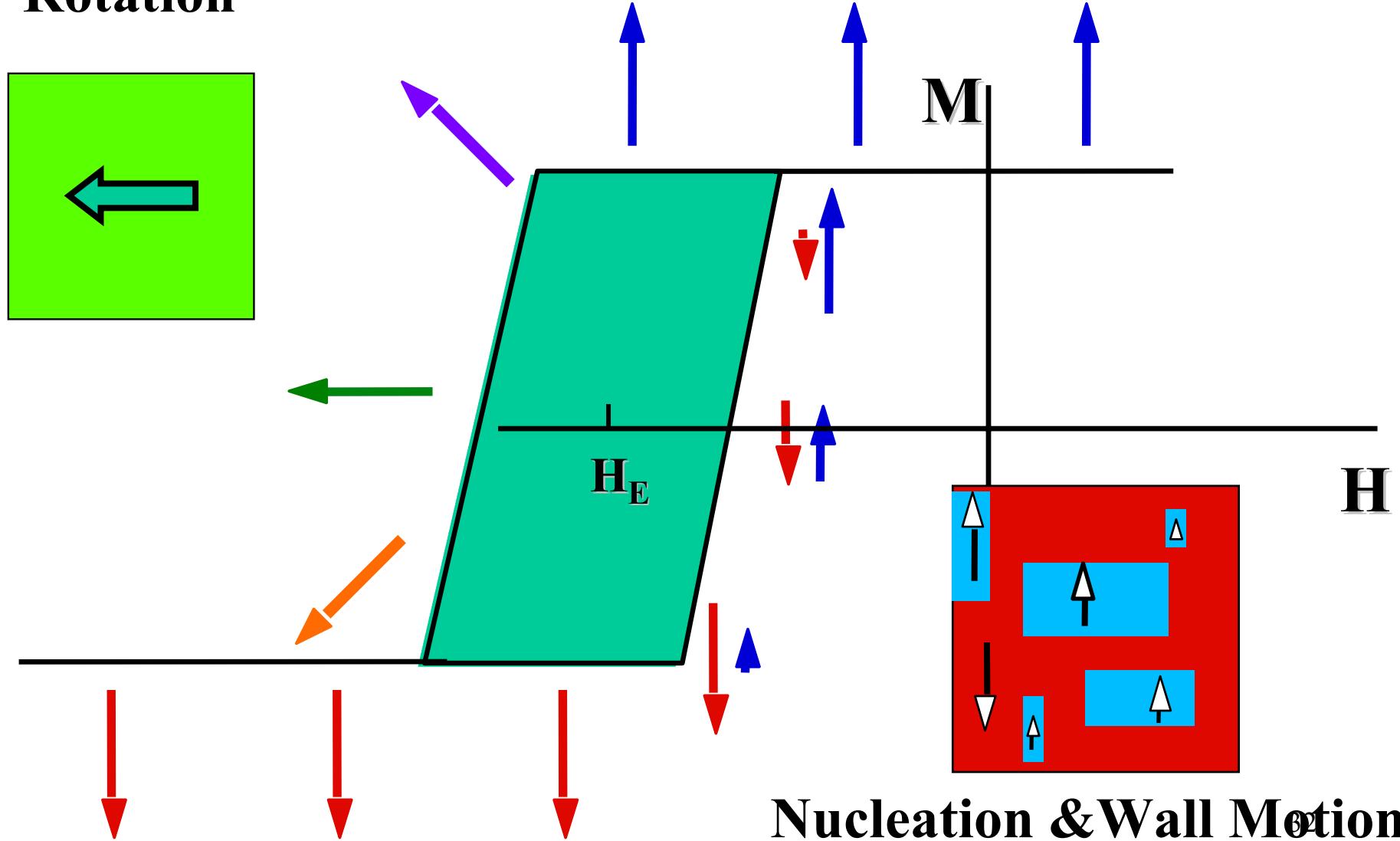
# Asymmetric reversal



# Asymmetric reversal

⇒ Largest  $H_E$

Rotation



Nucleation & Wall Motion

# Fe on *twinned* single crystal AF

- Asymmetric magnetization reversal
- Asymmetry correlated with  $H_E$ .
  - Cooling field
  - Temperature
- Frustration of perpendicular exchange

M.R. Fitzsimmons et. al., Phys. Rev. Lett., 84, 3986 (2000)

# SUMMARY

- **Burried Interfaces**

M and Non-M Roughness-*Different*

- **Nanoscopic Magnetic Ordering**

Magnetic Orientation in Multilayers-*Delicate*

- **Vector Magnetometry**

Exchange Bias in Bilayers-*Complex*

**Do this with synchrotrons,  
Watson !!!**

**Sherlock Holmes**

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# Future

## Neutron

- Diffuse
- Inelastic
- Time dependence
- Pump-Probe

## Systems

- Burried interfaces
- Multilayers
- Superlattices
- Nanostructures

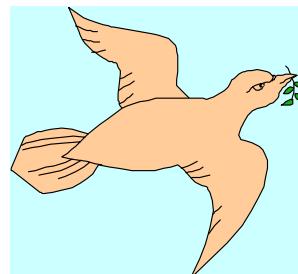
## Sample environment

- H field
- Low temperature
- High pressure

**BEYOND PROOF OF CONCEPT**

# LESSONS

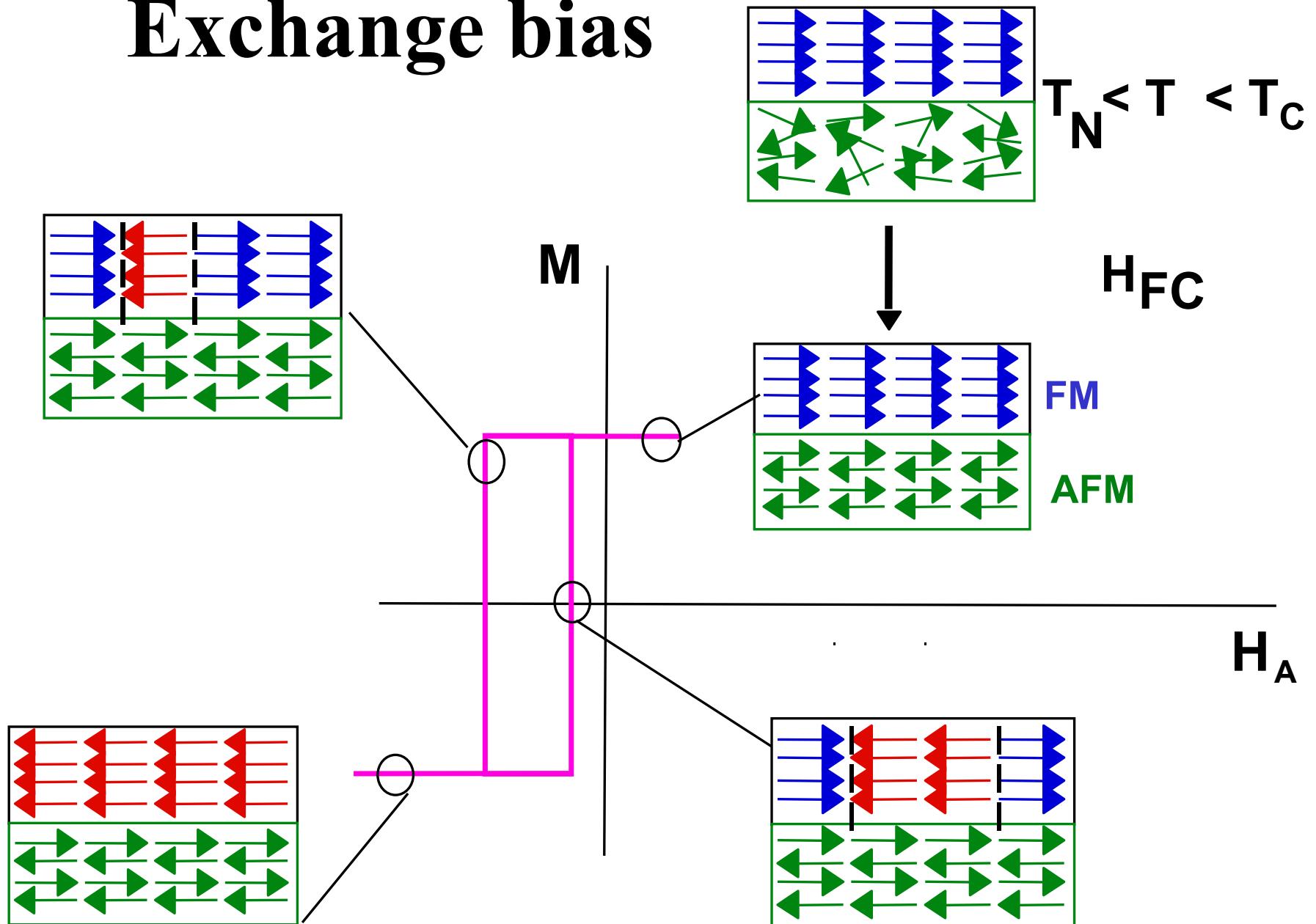
- Interpretation of neutron scattering straight forward
- Penetration Large
- Physical structure doesn't have all the information
- Magnetic structure subtle
- Need interesting materials and physics



Is OK

**Fini-End-Fin-Ende**

# Exchange bias



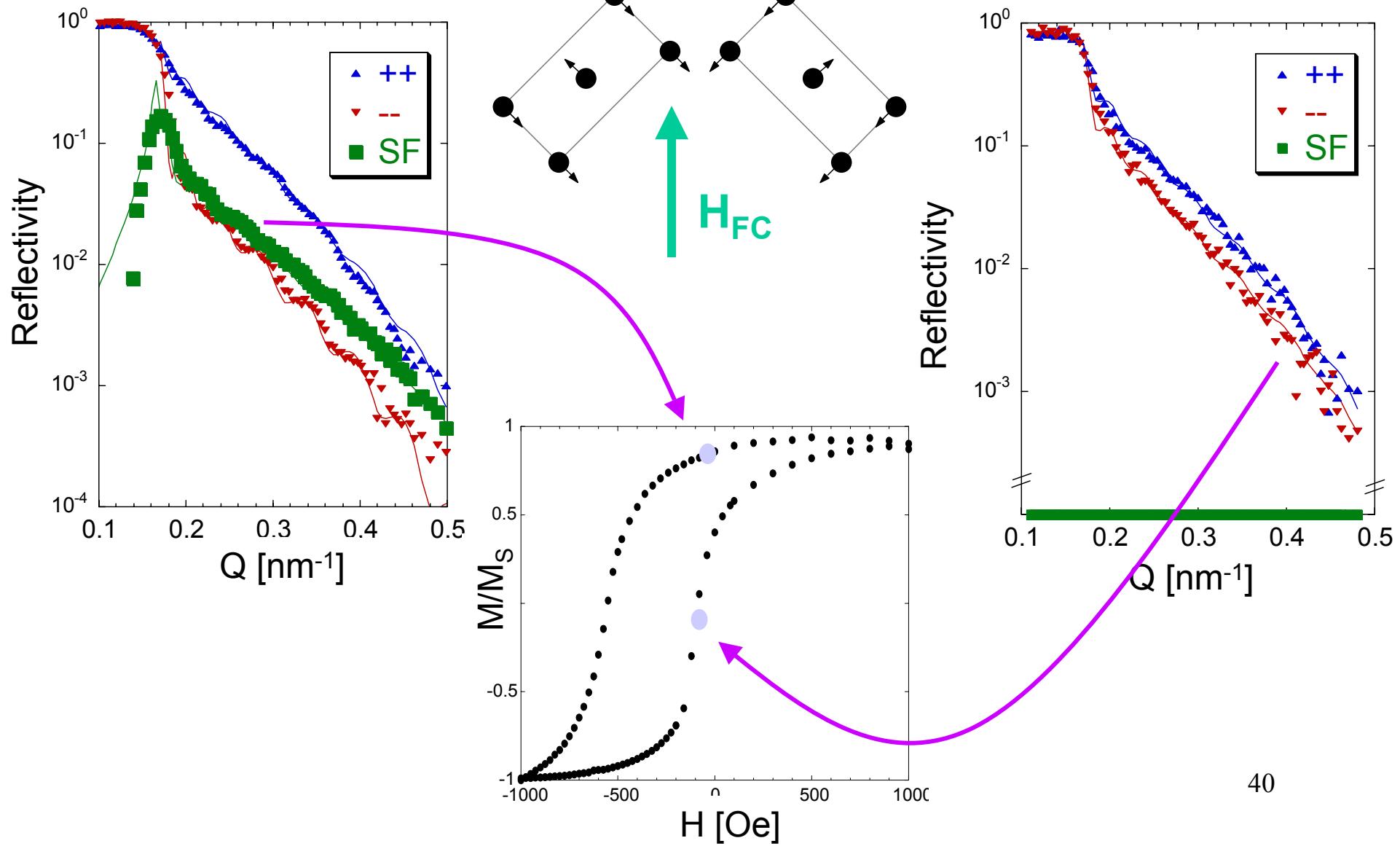
W.H. Meiklejohn, C.P. Bean, Phys. Rev., 105, 904<sup>38</sup>(1957).

# Approach

- **Model systems**
  - Controlled conditions.
  - Tailored epitaxy (crystallography).
  - Tailored chemistry (pure and doped films).
  - Tailored microstructure (interface roughness, twinning)
- **Experimental Tools**
  - SQUID
  - X-ray diffraction (high and low angle)
  - Ferromagnetic resonance
  - Magnetoresistance

**Large collaborative team effort**

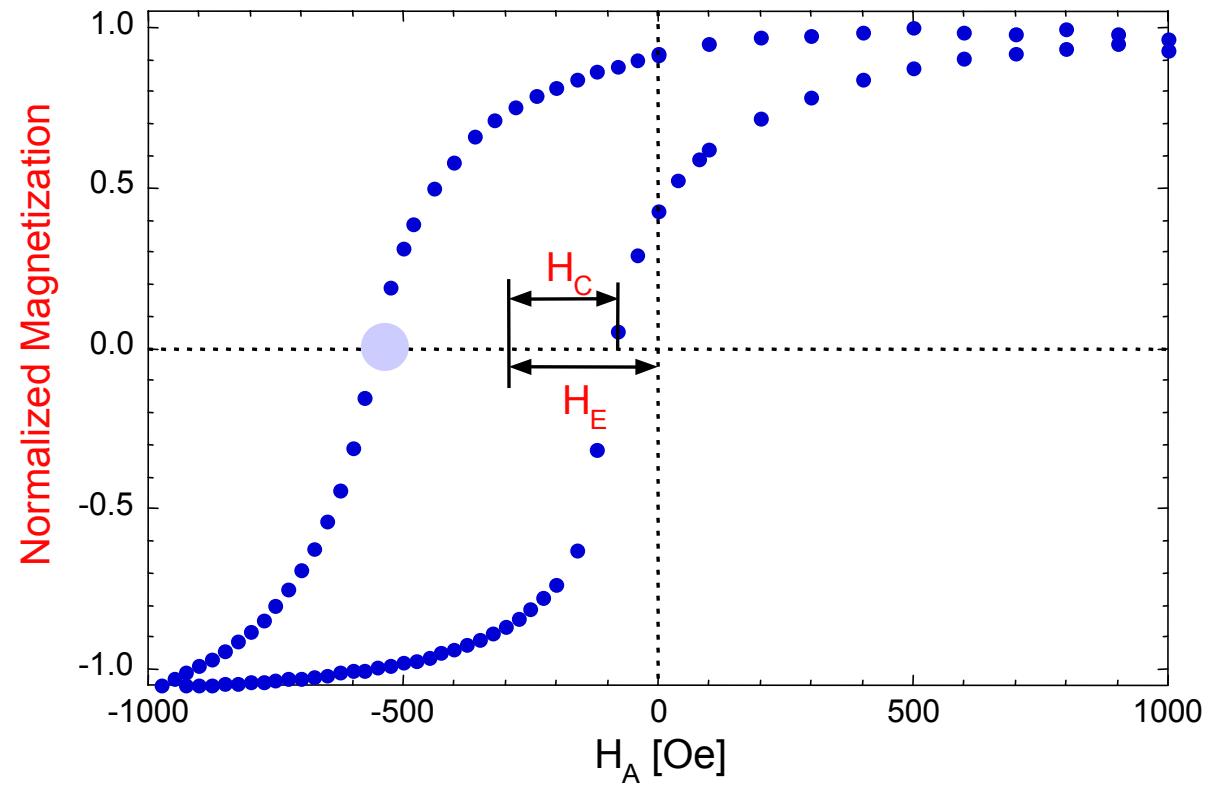
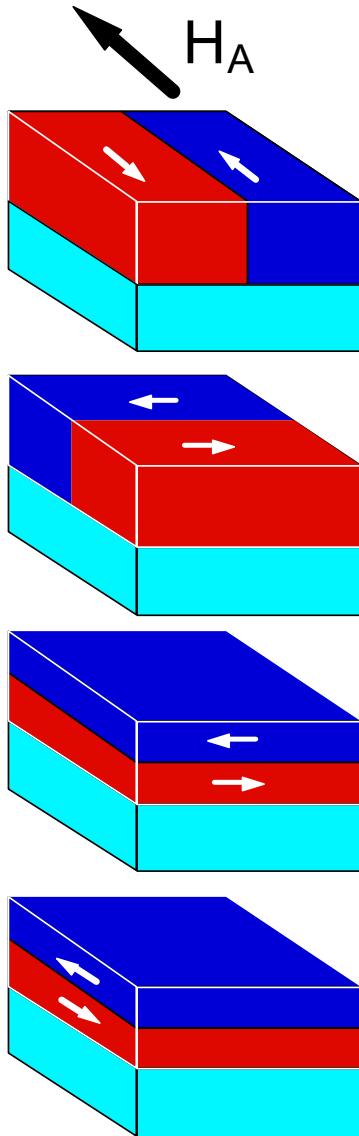
# Fe- *twinned* FeF<sub>2</sub>, case for H<sub>E</sub> = - 325 Oe.



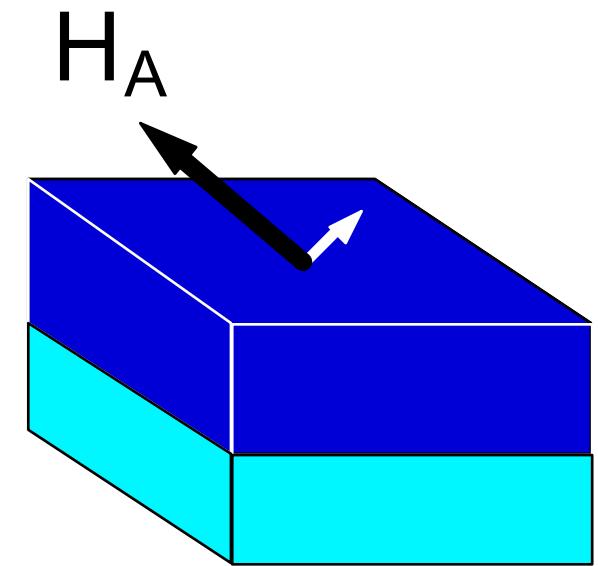
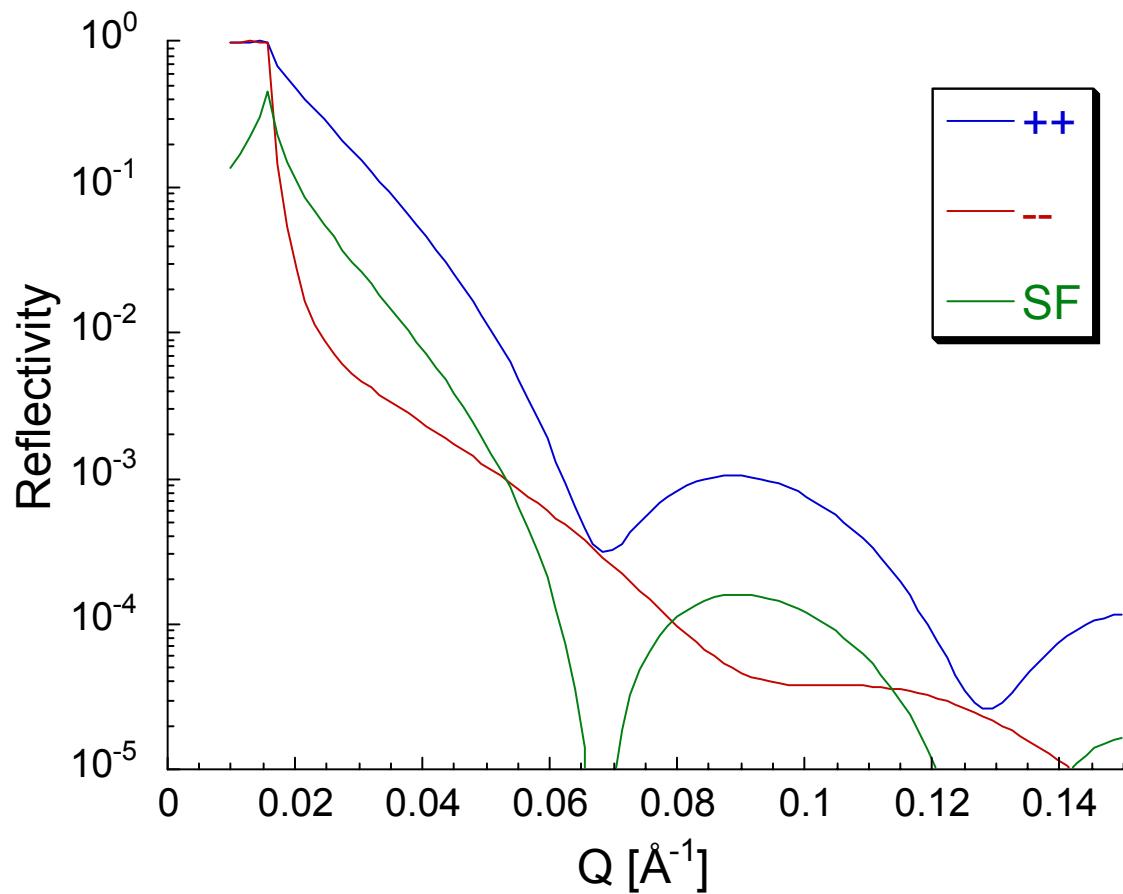
# Conclusions

- To enhance exchange bias:  
$$\sum_{\text{domains}} \left| \vec{S}_F \bullet \vec{S}_{AF} \right| \neq 0$$
  - Choose while field cooling.
  - Promote multiple well-defined anisotropy axes in FM.
  - Promote asymmetric magnetization reversal.

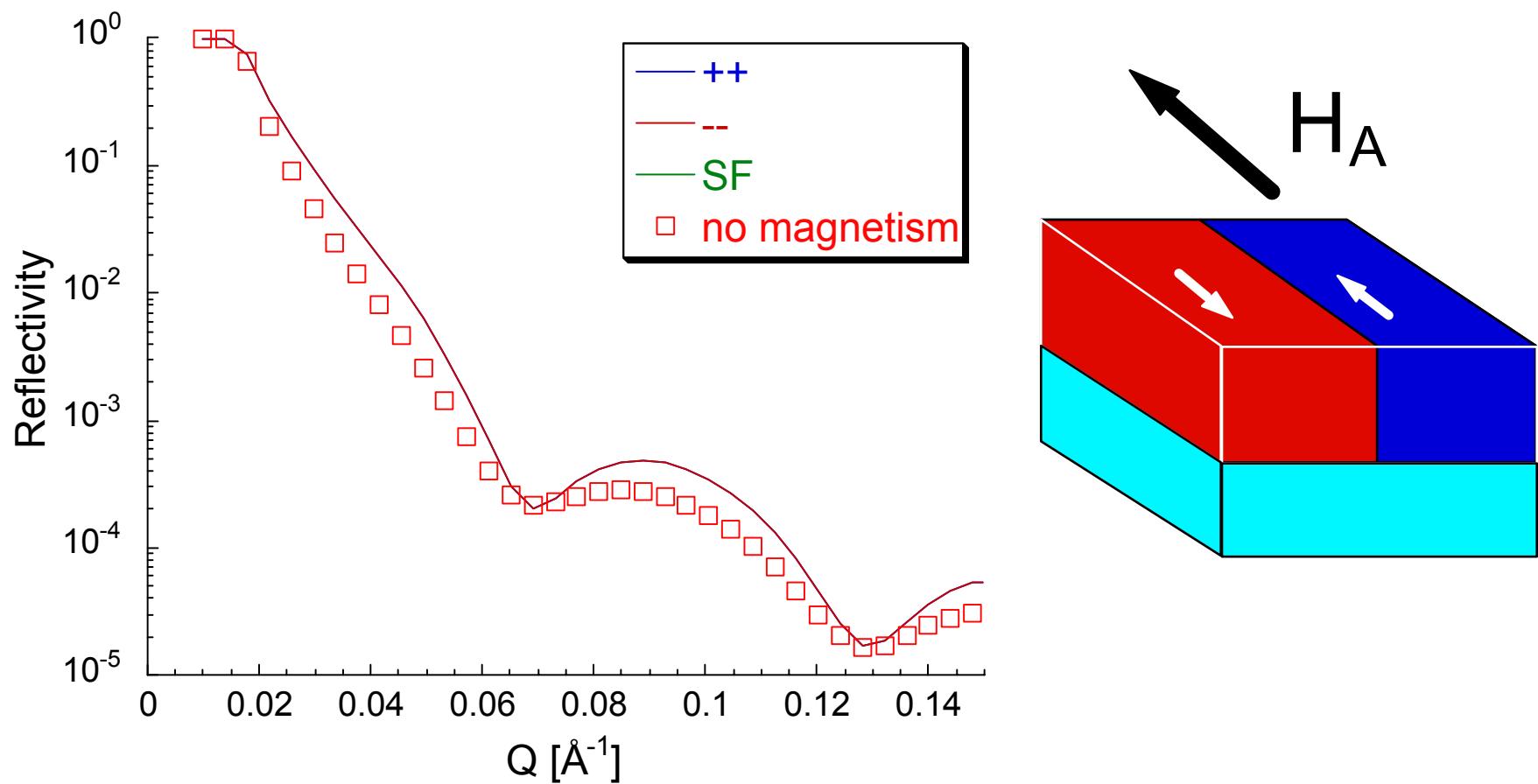
# Reversal process?



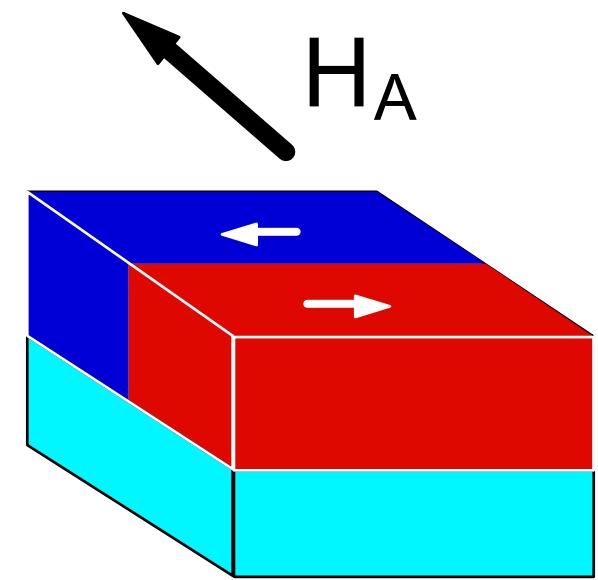
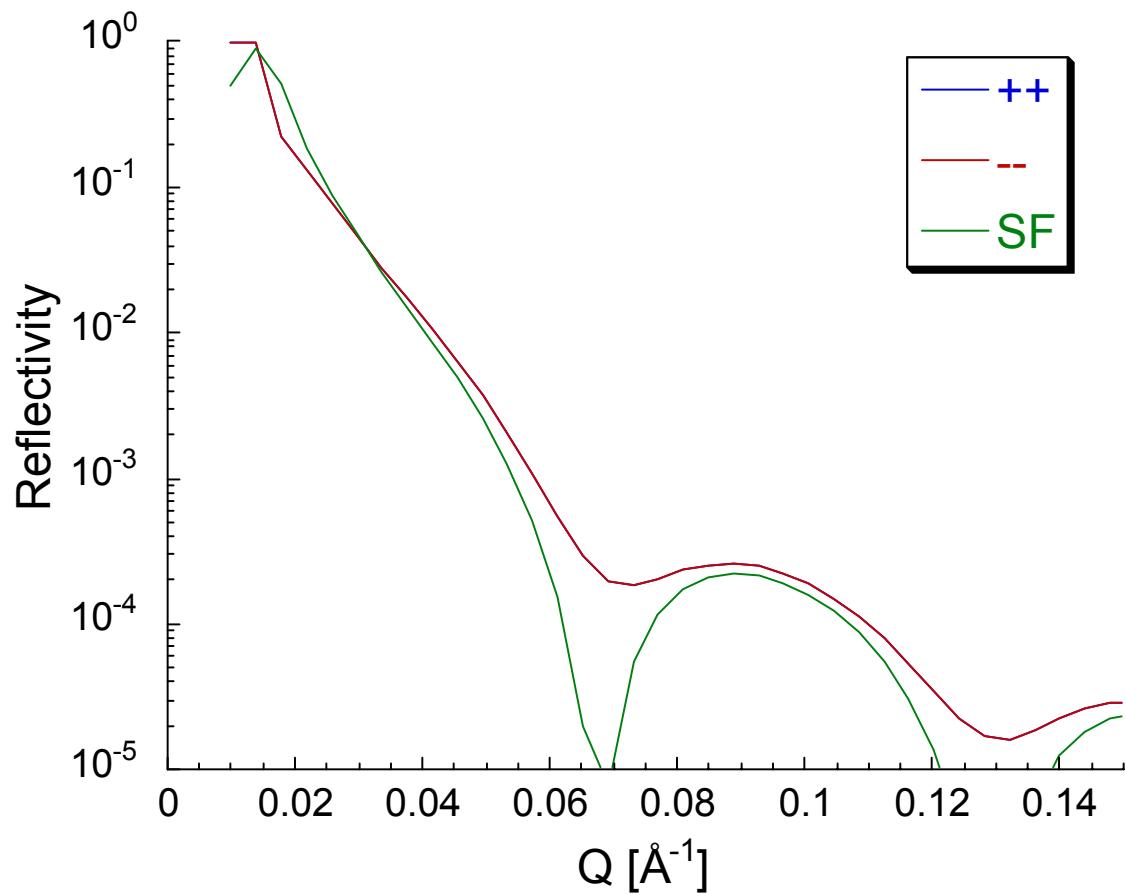
# Simple example for coherent rotation



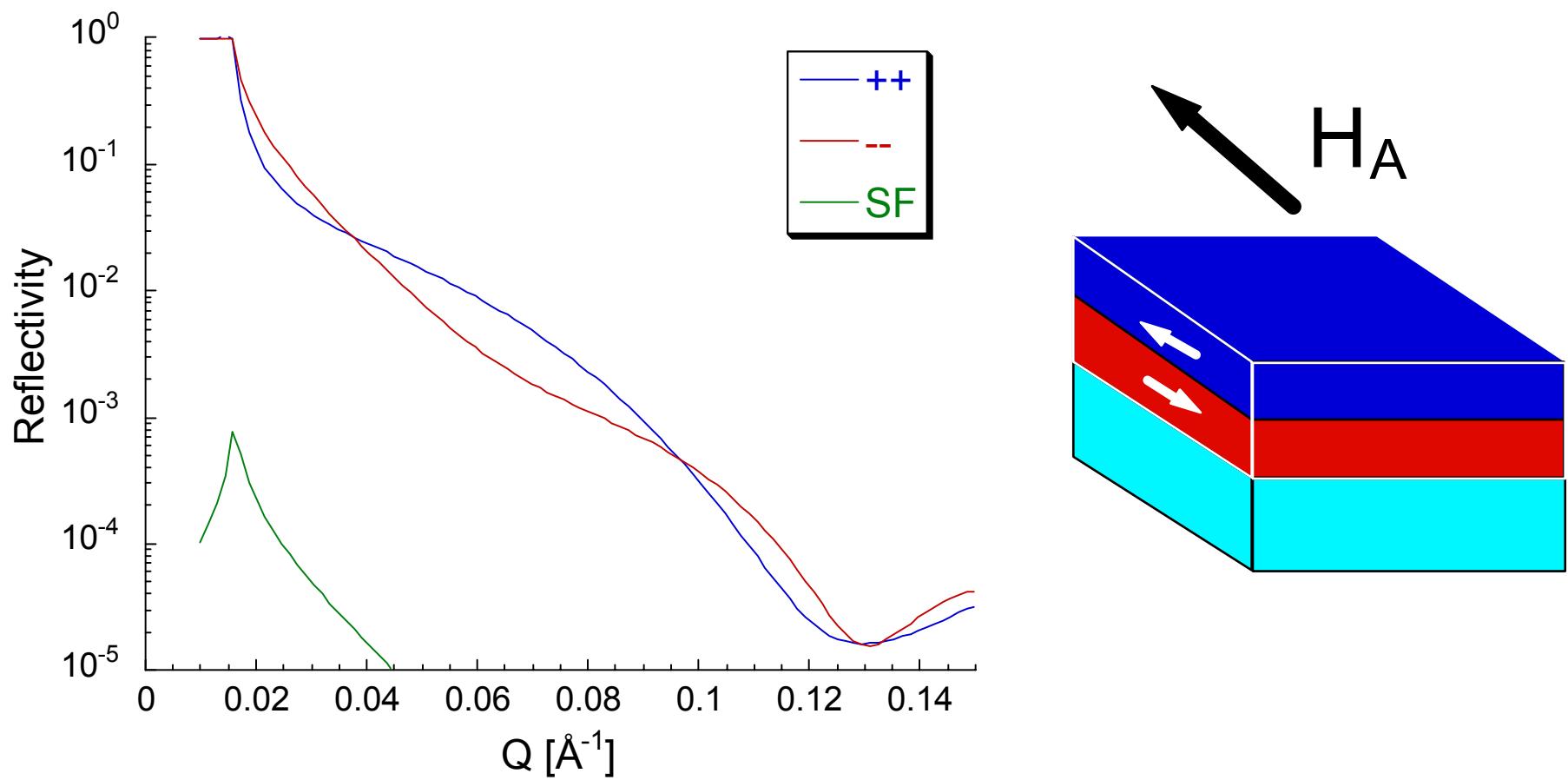
# Example 1: Opposed domains



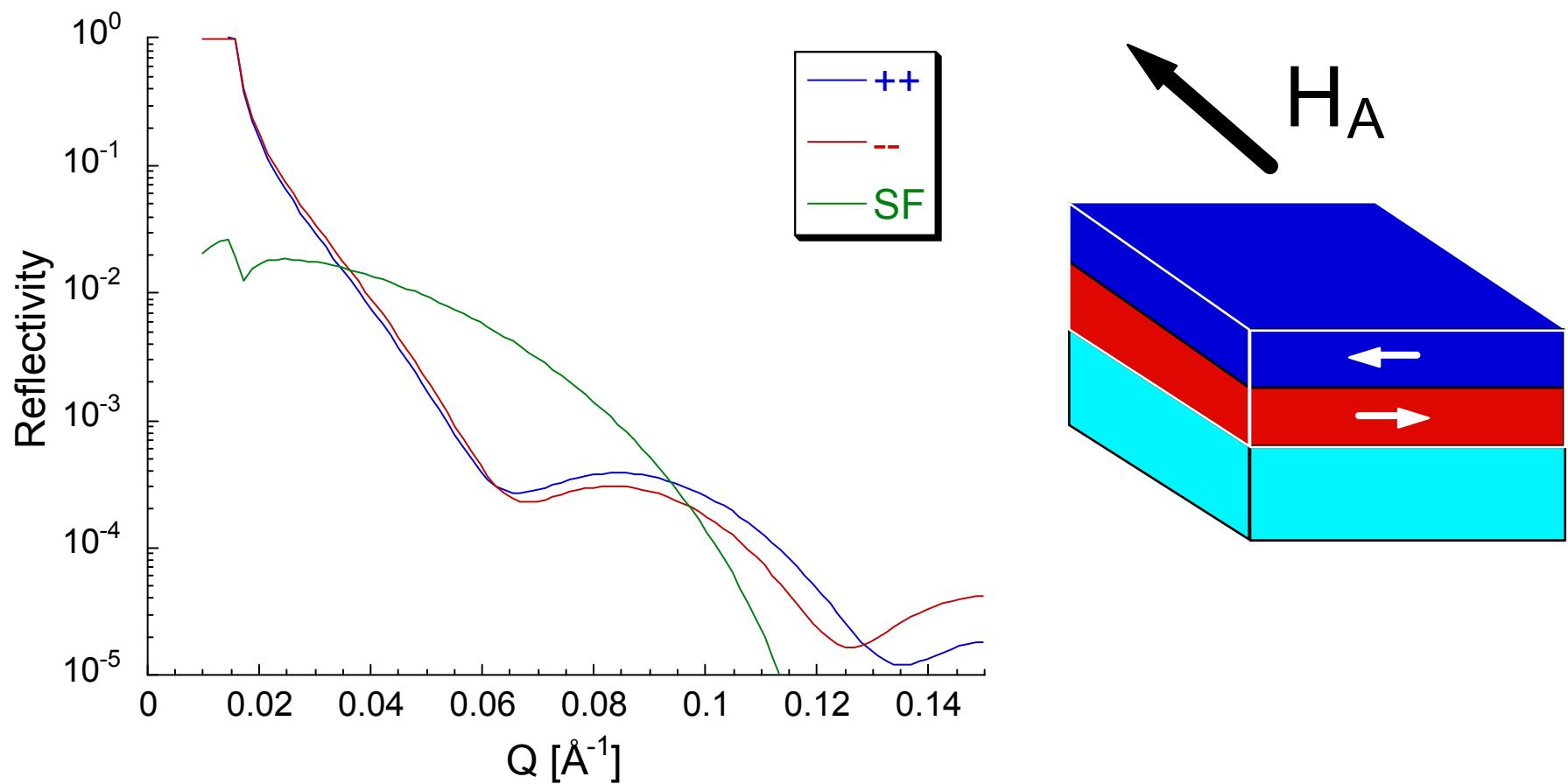
## Example 2: Opposed domains + rotation



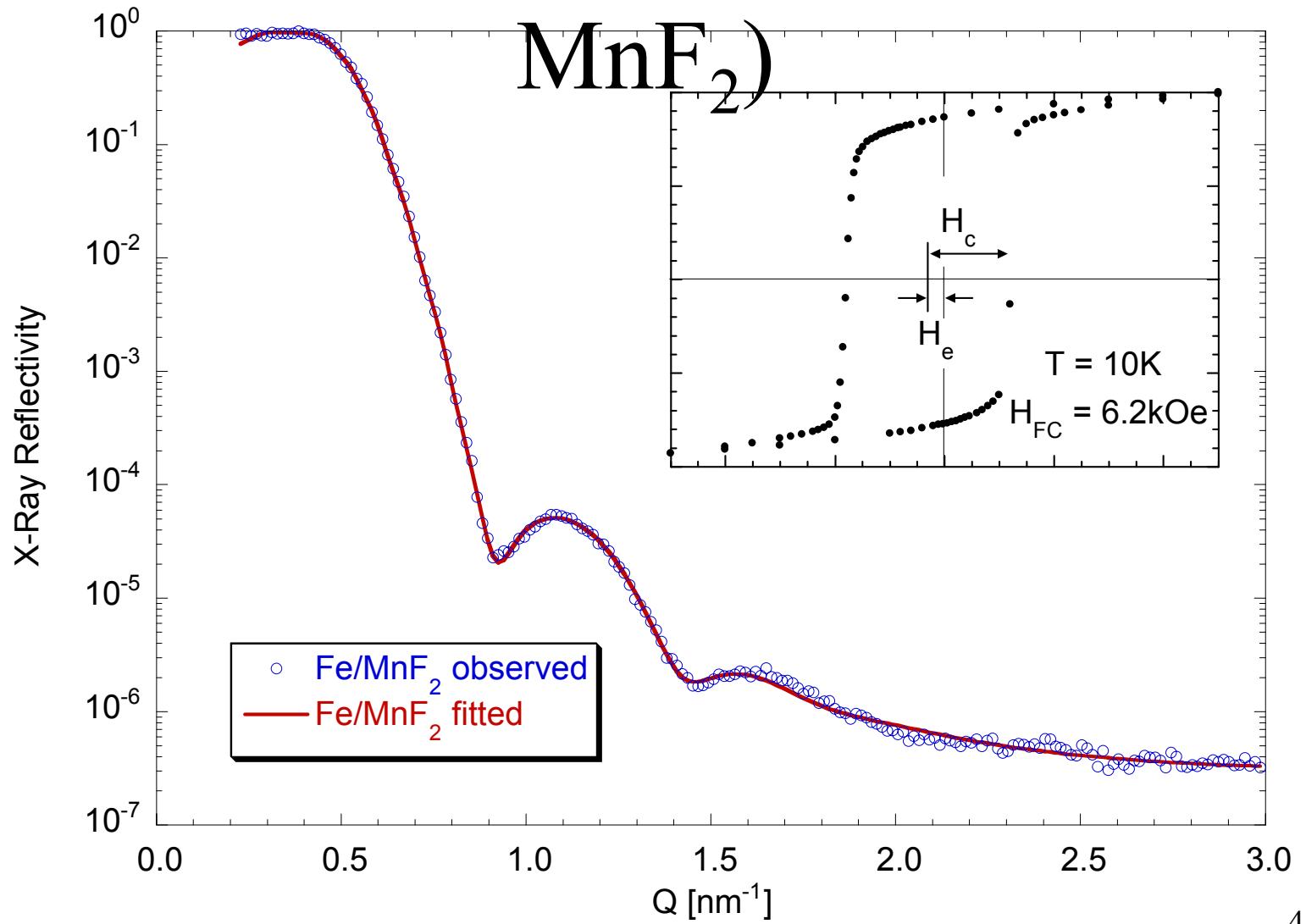
# Example 3: Layered domains



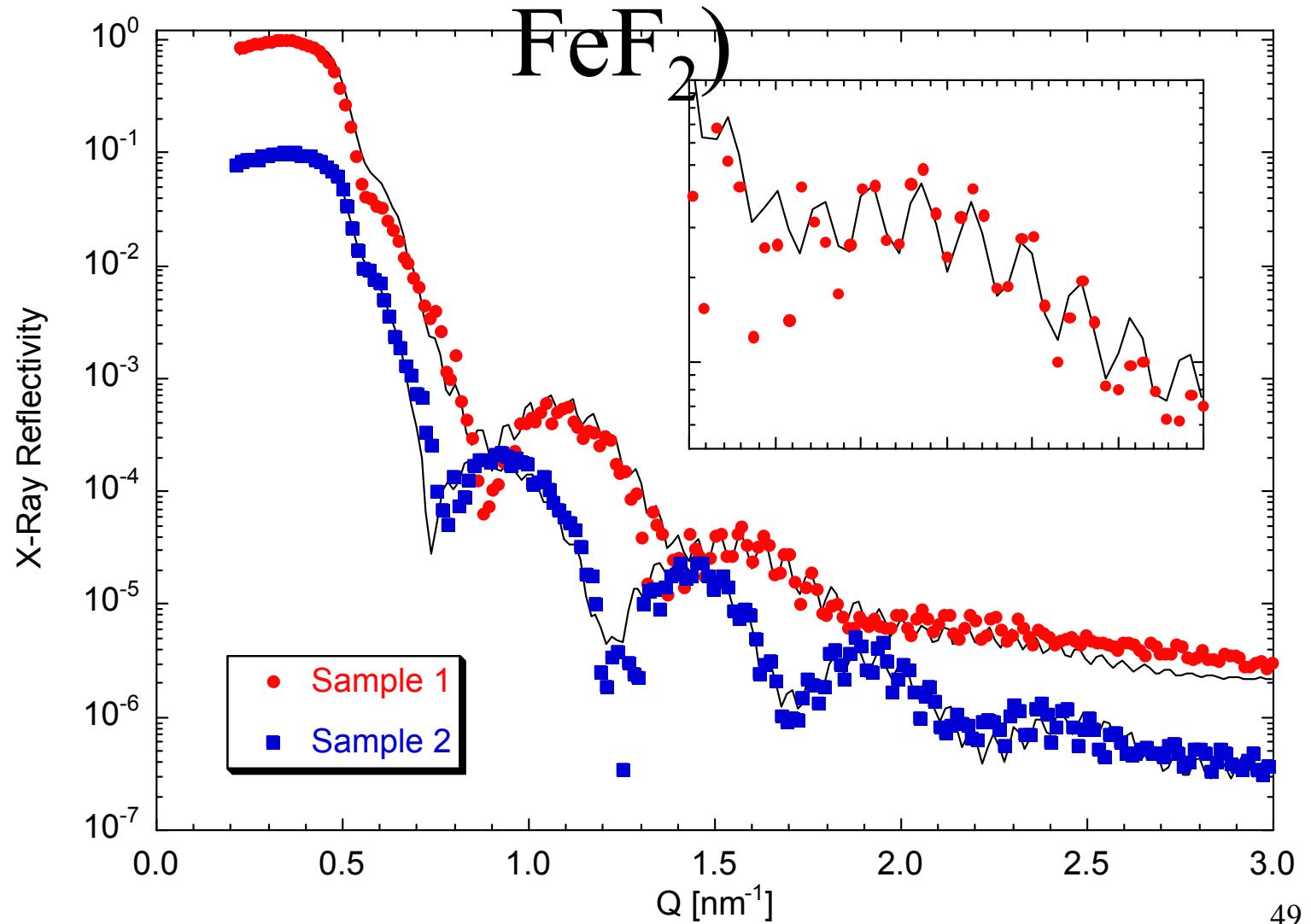
# Example 4: Layered domains + rotation



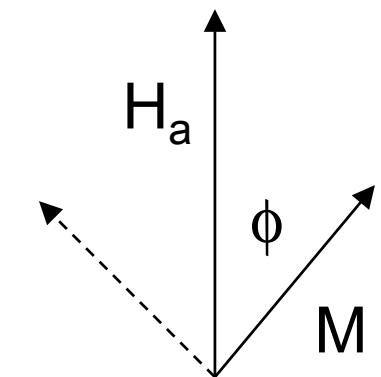
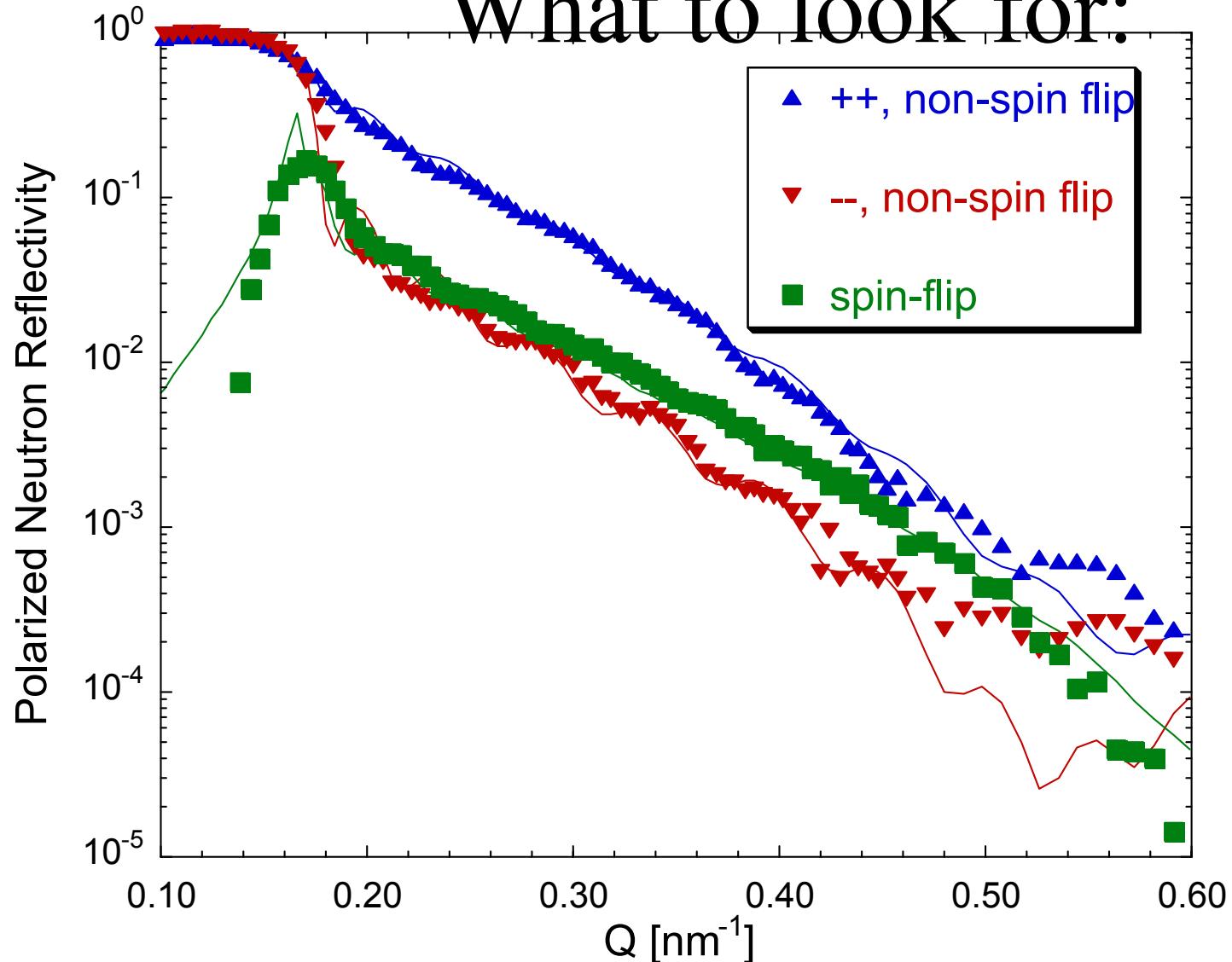
# X-ray reflectometry (Fe on MnF<sub>2</sub>)



# More X-ray reflectometry (Fe on

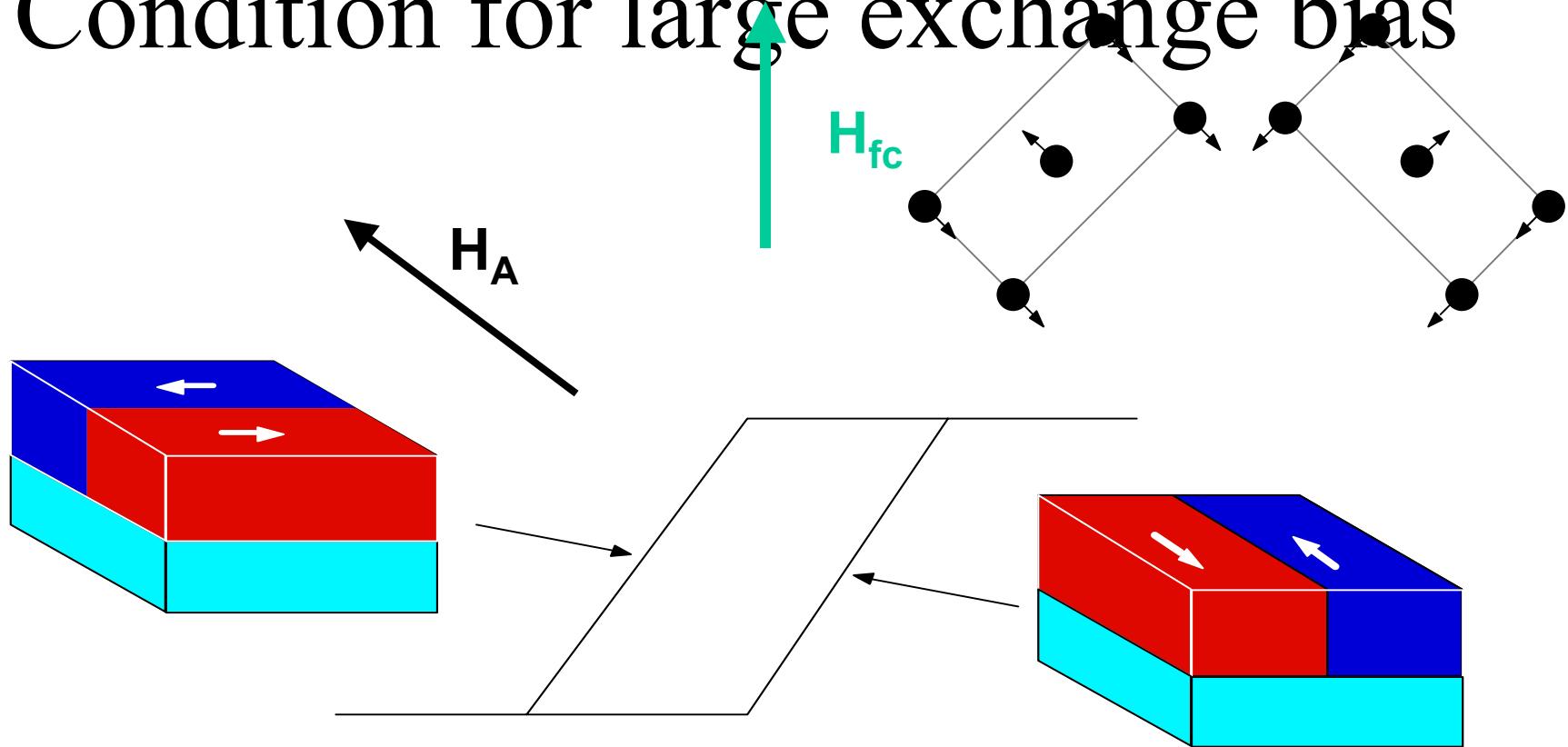


# What to look for:



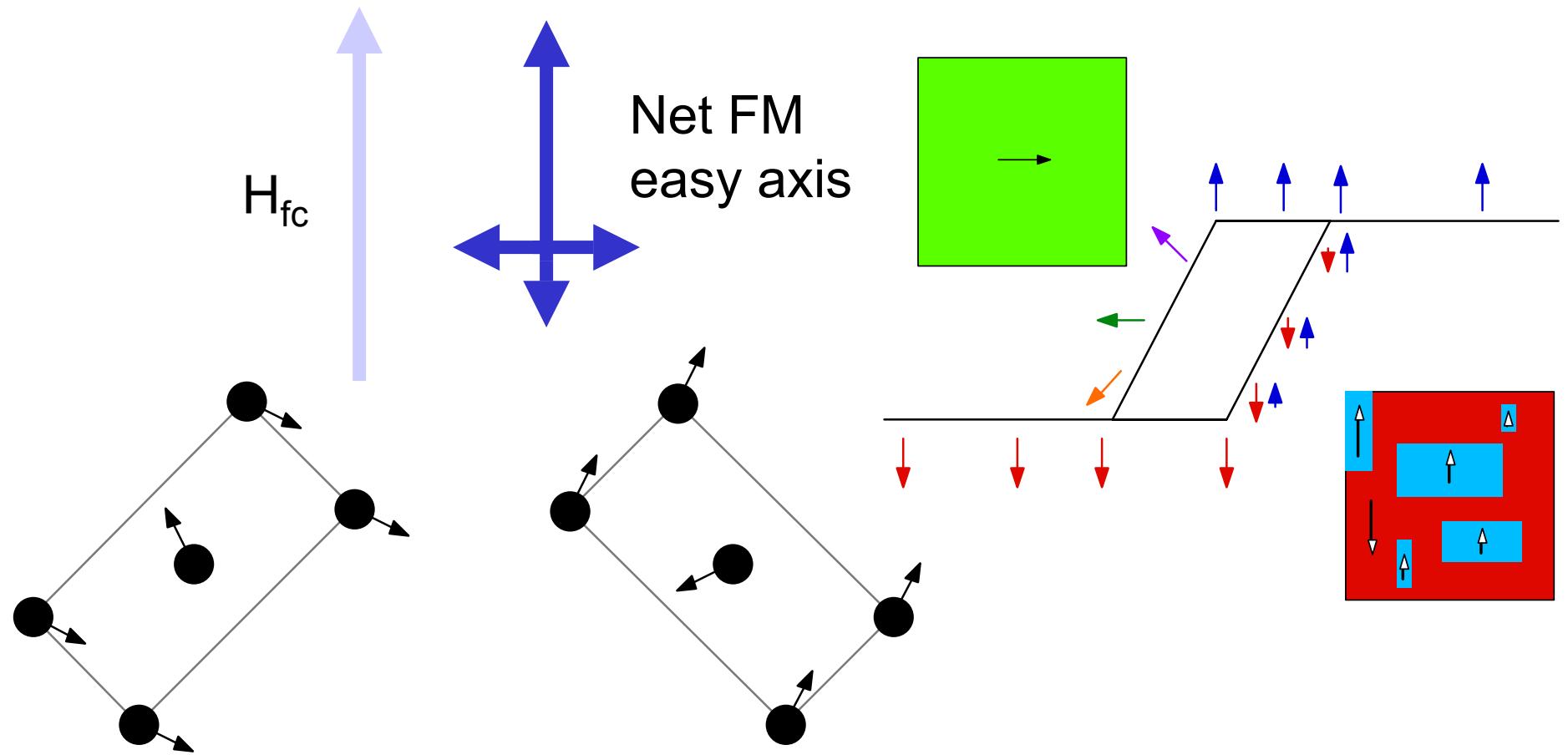
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# Condition for large exchange bias



# Why does the asymmetric reversal process occur?

**Large exchange bias**



# More conclusions

- Spin-flip neutron scattering is a quantitative measure of magnetization rotation away from applied field.
- SF scattering for  $T < T_N$  varies as  $B_{5/2}(T)$  for a smooth sample, but not for a rough sample.
- T-dependence of SF scattering (and asymmetric magnetization reversal) correlates with  $H_E$ .
- Uniaxial anisotropy axis perpendicular to the unidirectional anisotropy axis is an important

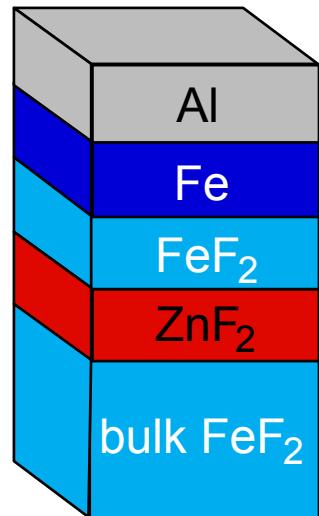
# Yet more conclusions

- Large asymmetry in reversal process is correlated with large  $H_E$ .
  - Cooling field (orientation) dependence.
  - Temperature dependence.
  - Cooling field (strength) dependence.

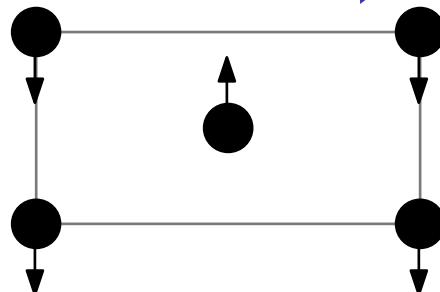
# Future directions

- Non-uniformity in magnetic heterostructures
  - Incomplete domain walls in exchange bias and exchange spring systems
  - Influence of chemical doping and/or geometrical frustration on FM & AFM
  - Confinement, e.g. suppression of domains in thin films
  - Incomplete domain walls and reorientation transitions, e.g. Fe(110)/W(110)

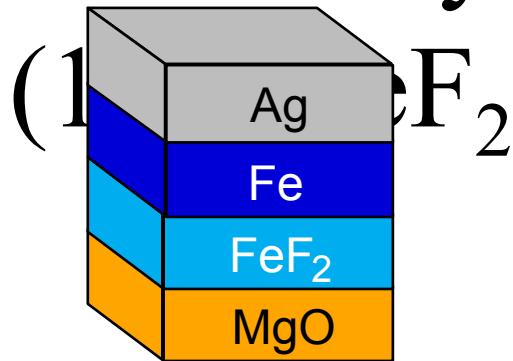
# A systematic study of Fe on



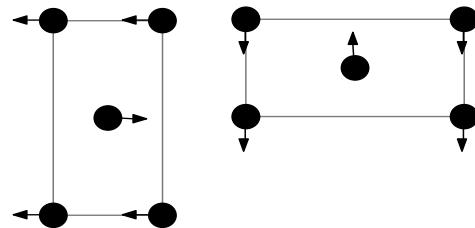
Untwinned



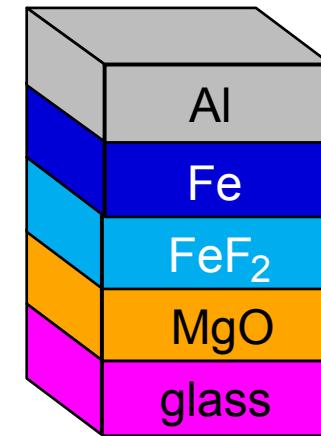
⊥- coupling  
not sufficient



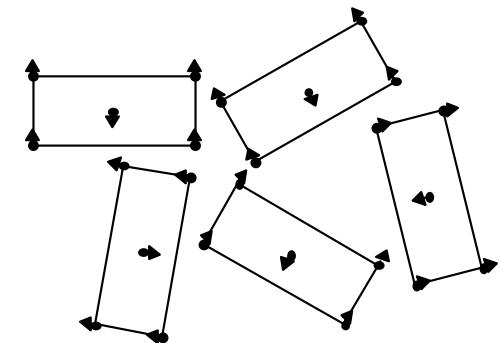
Twinned



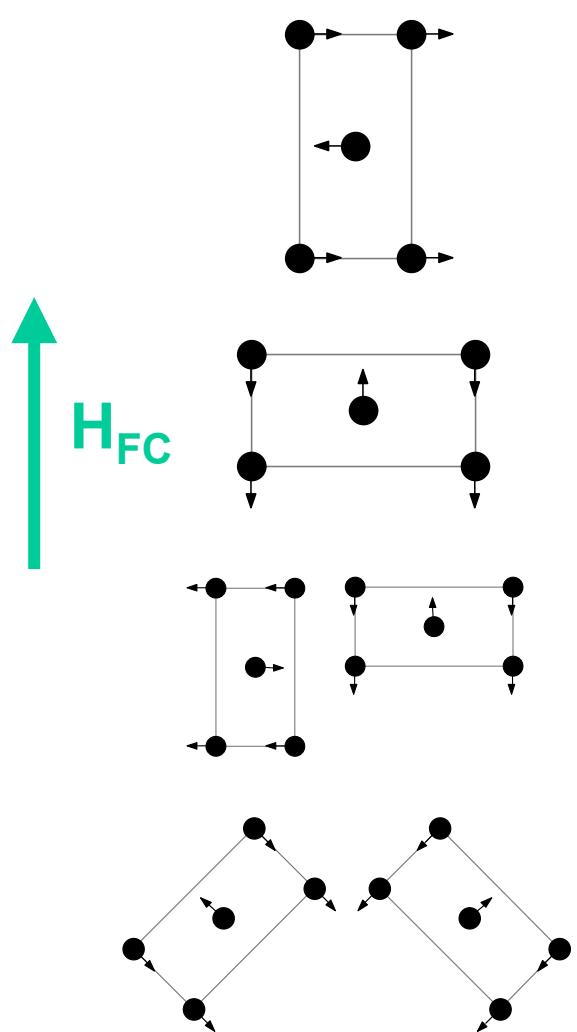
Asymmetrical  
reversal



In-plane  
polycrystalline



⊥- coupling  
not required<sup>6</sup>



Summary  
 $H_E \sum_{domains} |\vec{S}_F \cdot \vec{S}_{AF}|$

0

-32

-76

-325

$K_F$ 's



0

1

0.5

0.7



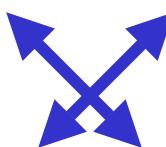
$SF \rightarrow$   $\leftarrow SF$

No

No

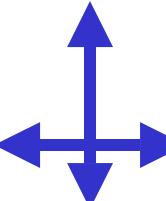
Yes

Yes



Yes

Yes



Yes

No

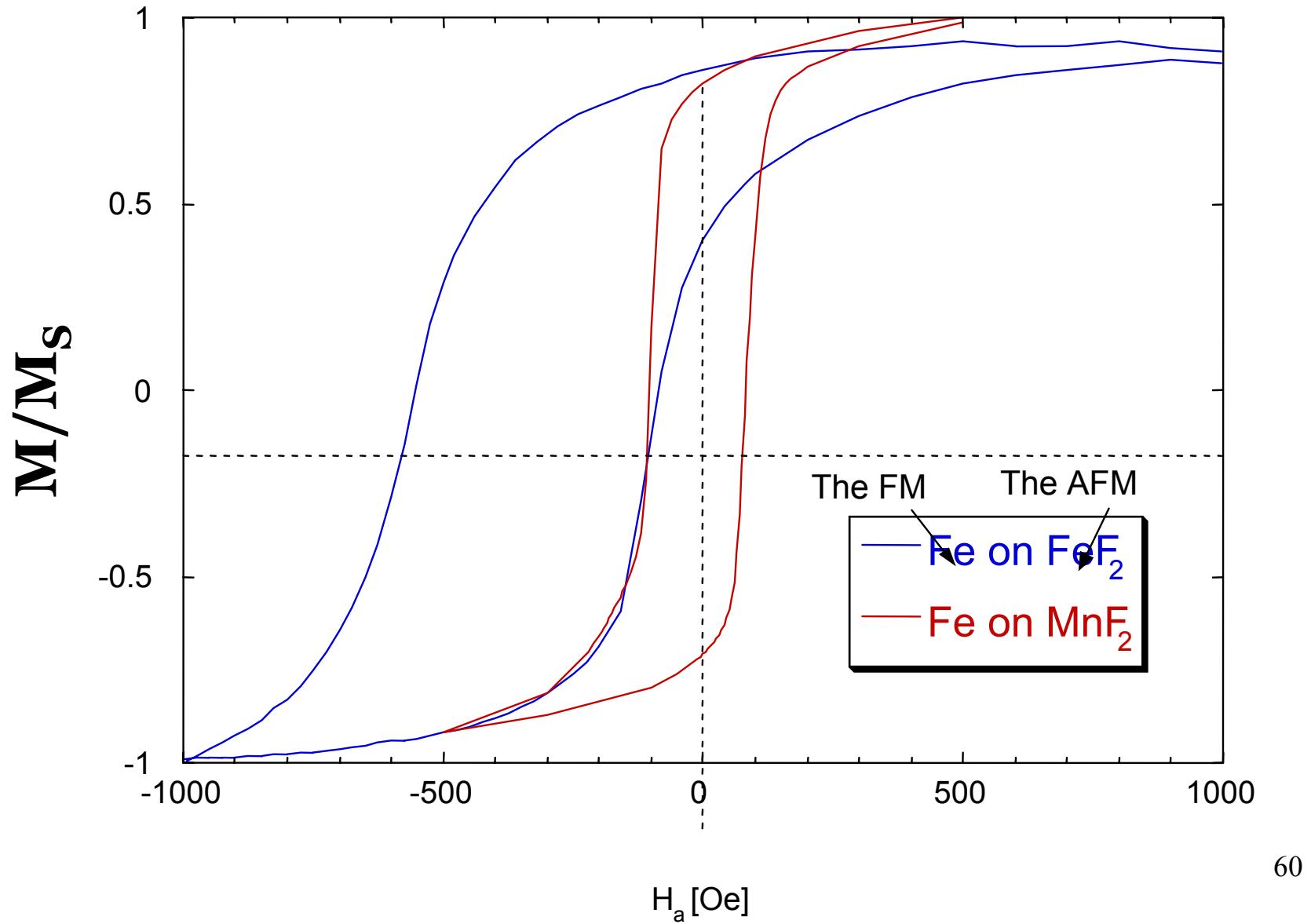
# Future

- Phonons....anomalous elastic constants
- Magnons.....unusual in thin films
- Modification by strain
- Burried interfaces
- Time Dependence
- Magnetic reversal
- Anisotropy
- Interfacial Magnetic Structure
- Diffuse Scattering (structure and magnetism)

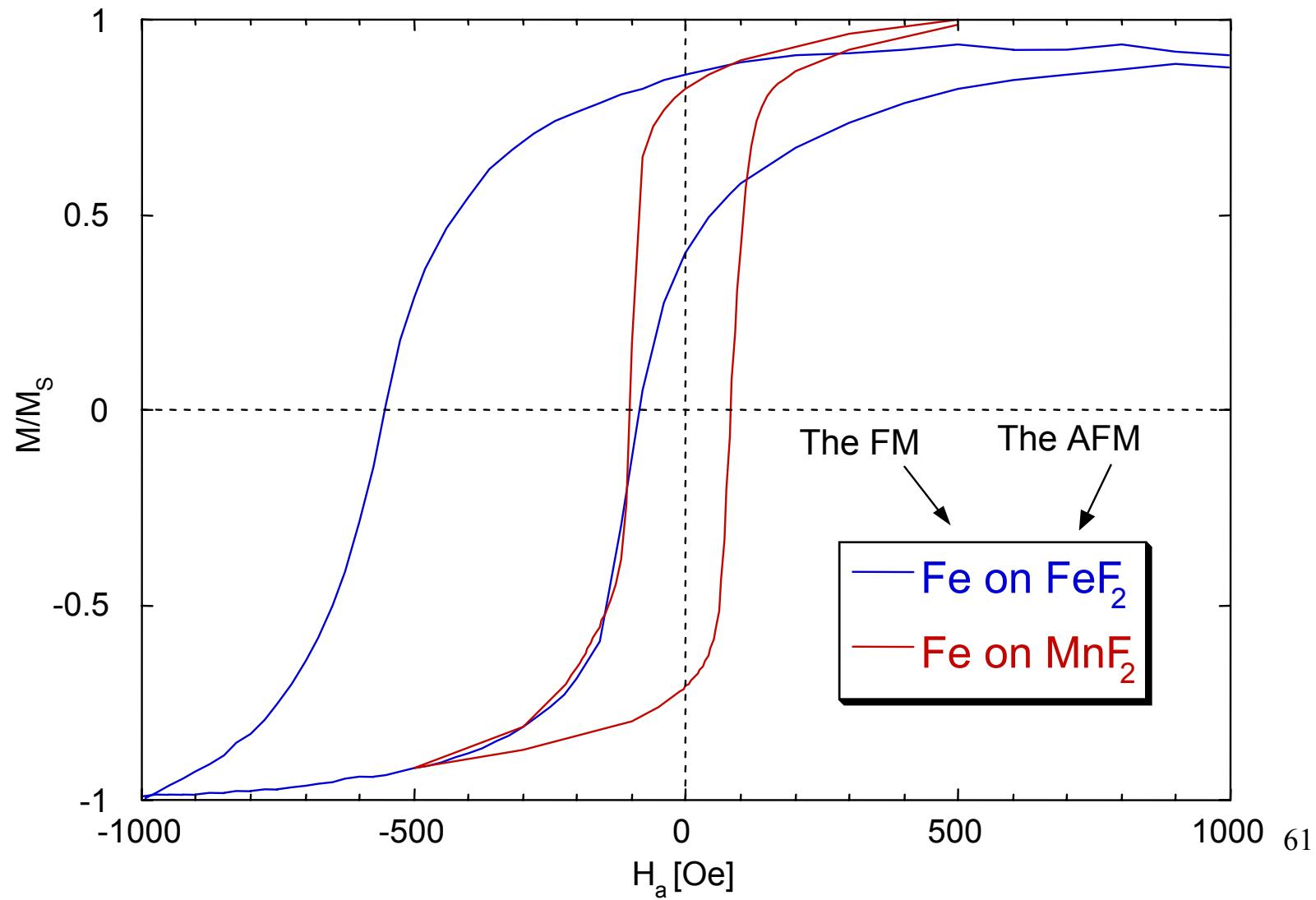
# What are some issues?

- Simple models incorrectly predict  $H_E$ .
- $H_E$  for *compensated* antiferromagnetic interfaces
- Positive  $H_E$
- T-dependence of  $H_E$  and  $H_C$
- $H_E$  and domains in ferromagnet or antiferromagnet
- Ferromagnetic moment in the antiferromagnet

# What is exchange bias?

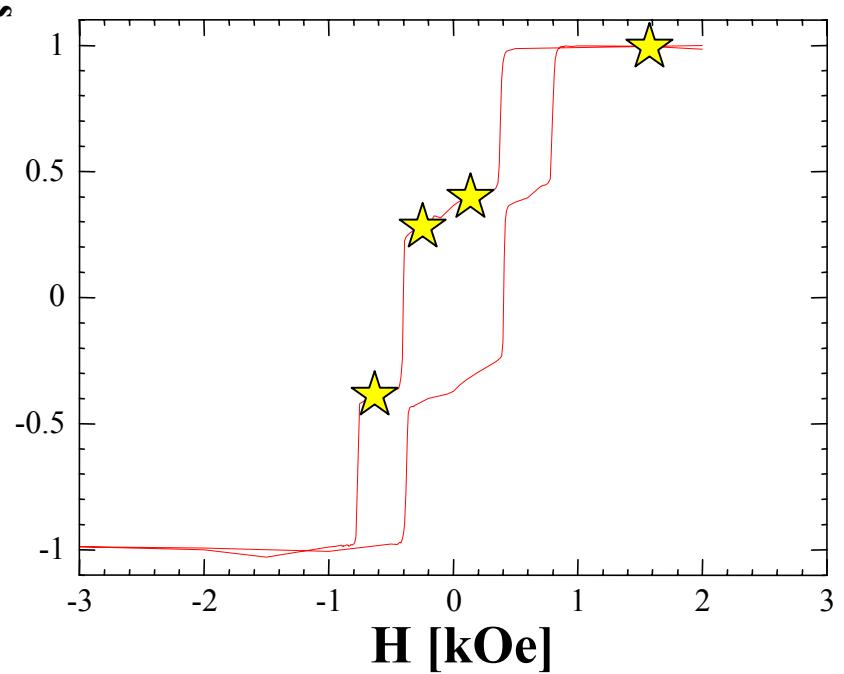
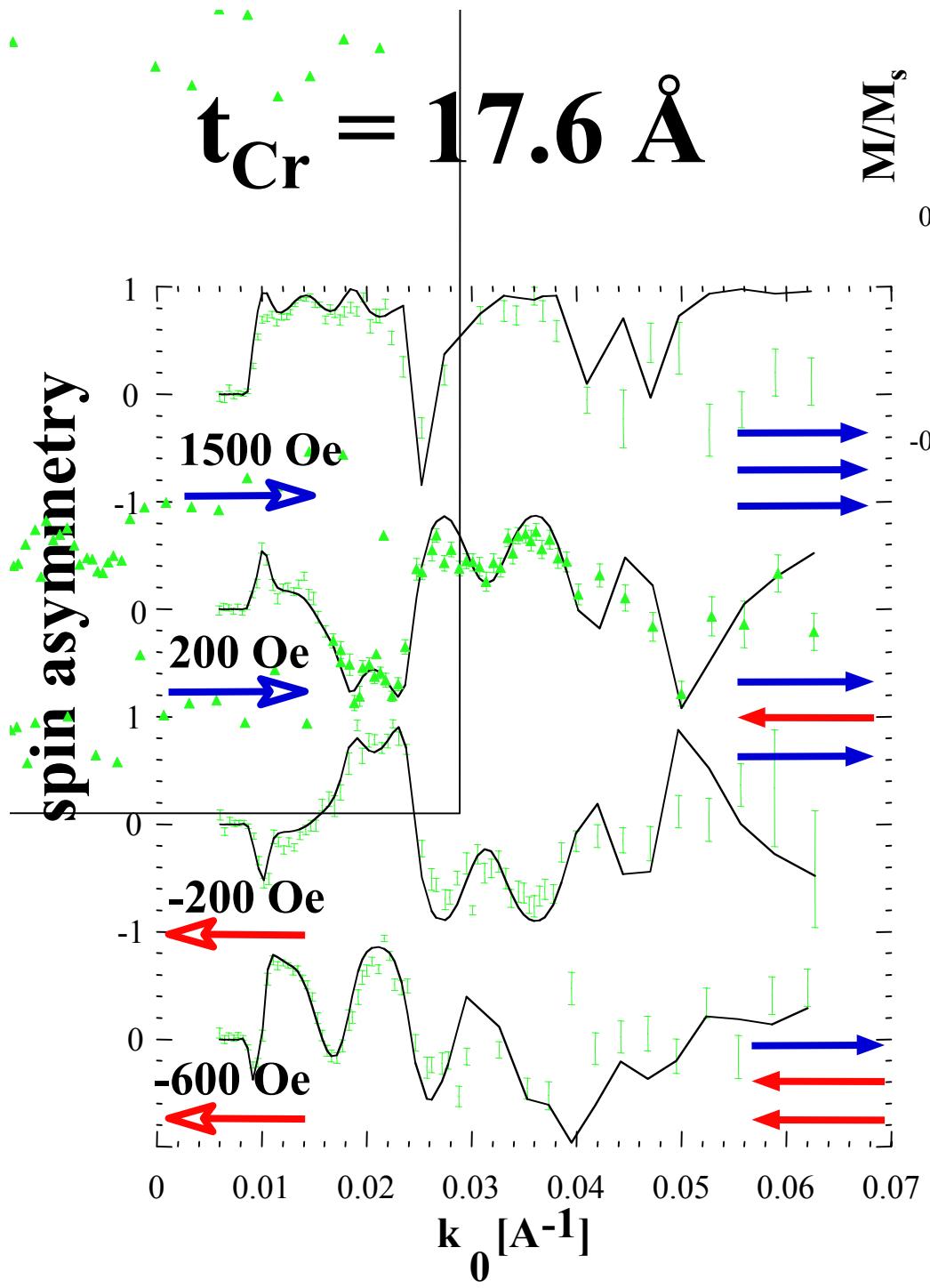


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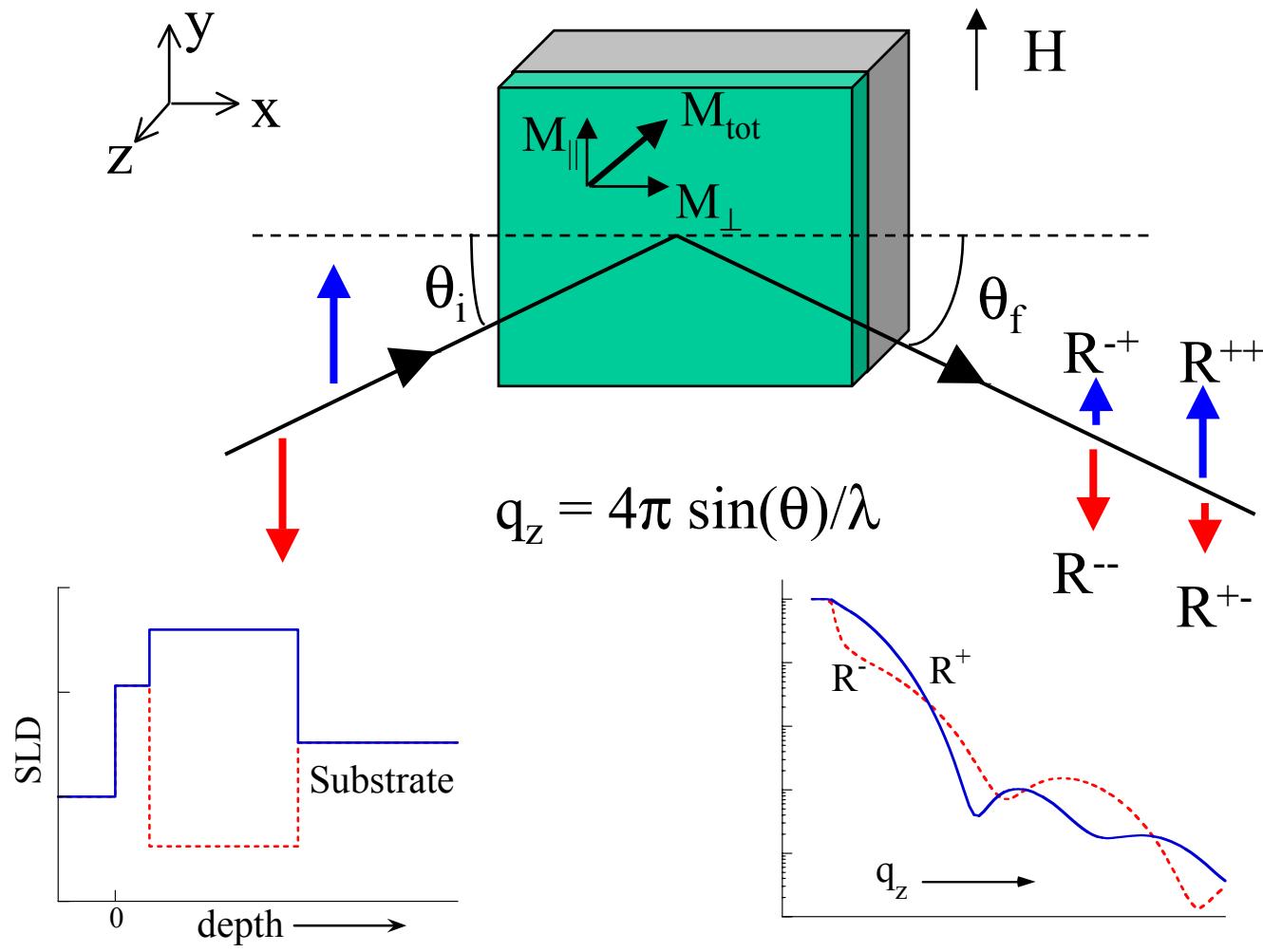
# What's our approach?

- Study model systems
  - Bilayers grown under controlled conditions.
  - Tailored epitaxy (crystallography).
  - Tailored chemistry (pure and doped films fabricated).
  - Tailored microstructure (interface roughness, twinning).
- Bring many investigative tools to bear
  - SQUID
  - Wide angle X-ray diffraction
  - Glancing incidence (in-plane) diffraction
  - X-ray reflectometry
  - Ferromagnetic resonance
  - Magnetoresistance
  - Magnetic interferometric optical imaging
  - SHMOKE
- Requires large collaborative team effort

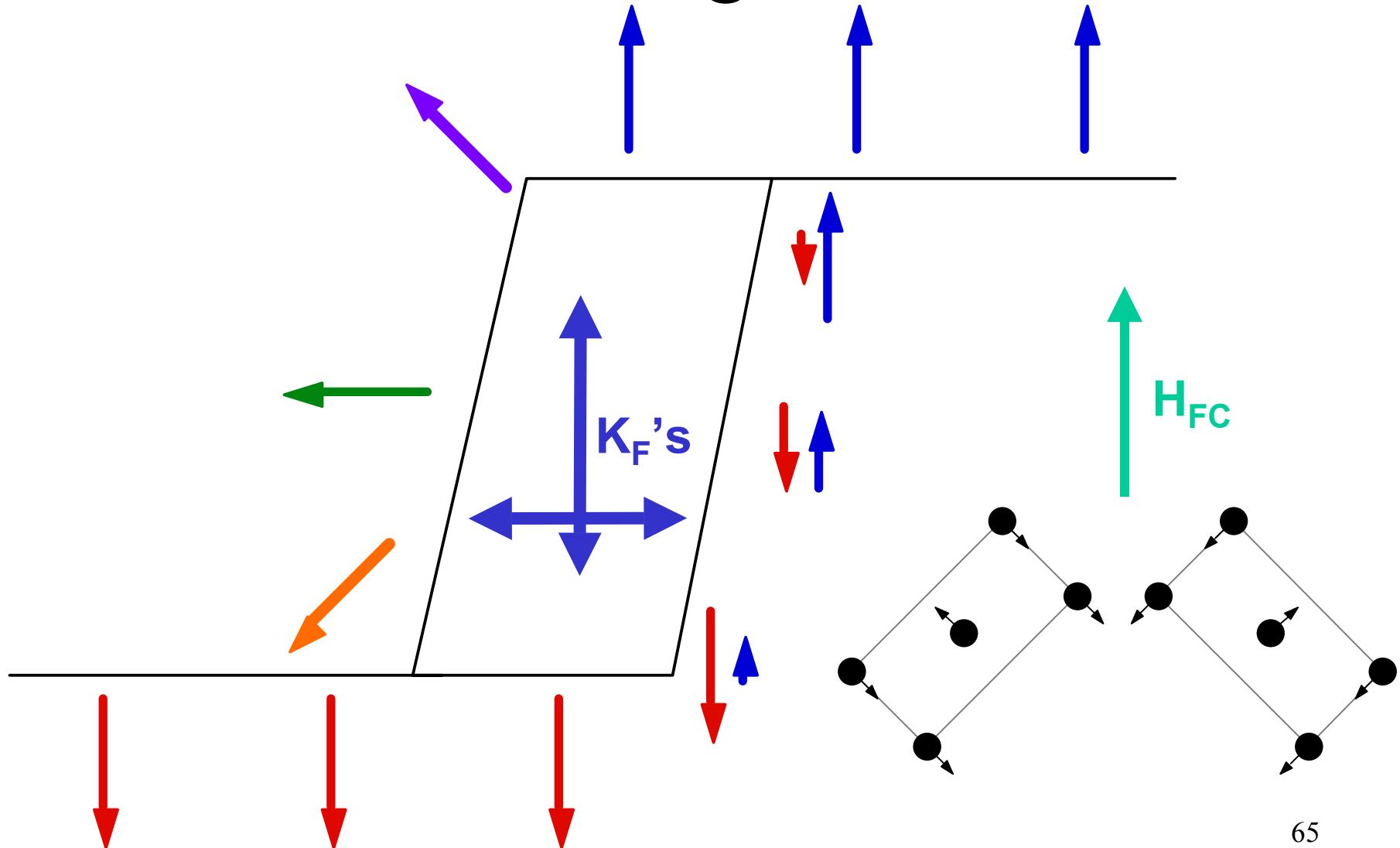


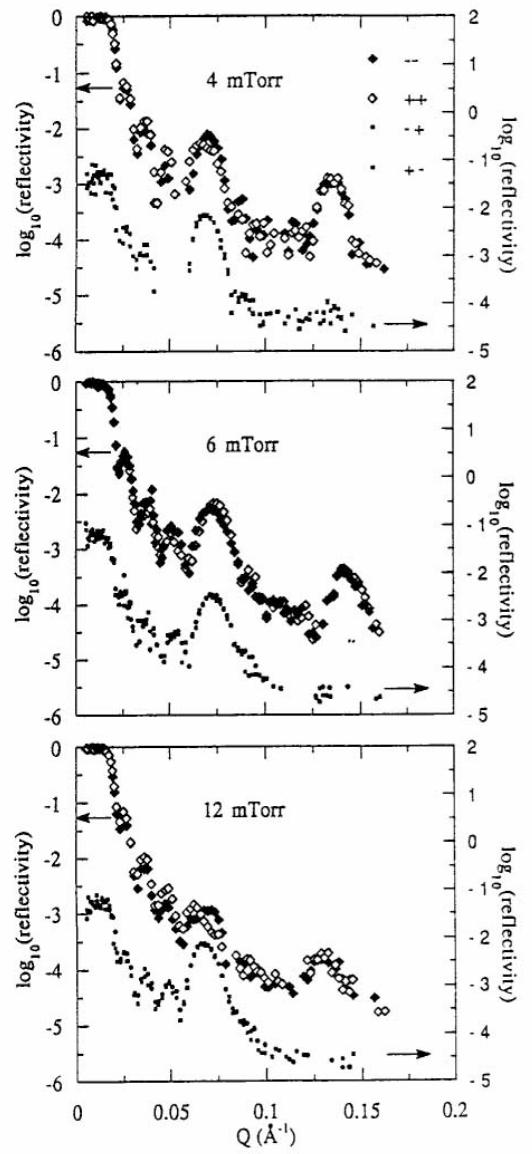
AFM coupling  
Top and bottom  
Different

# Polarized Neutron Reflectivity

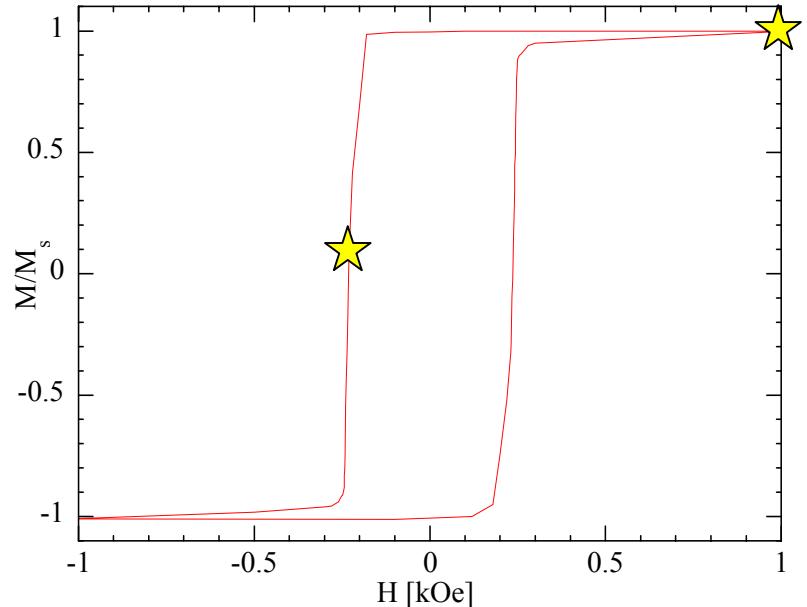
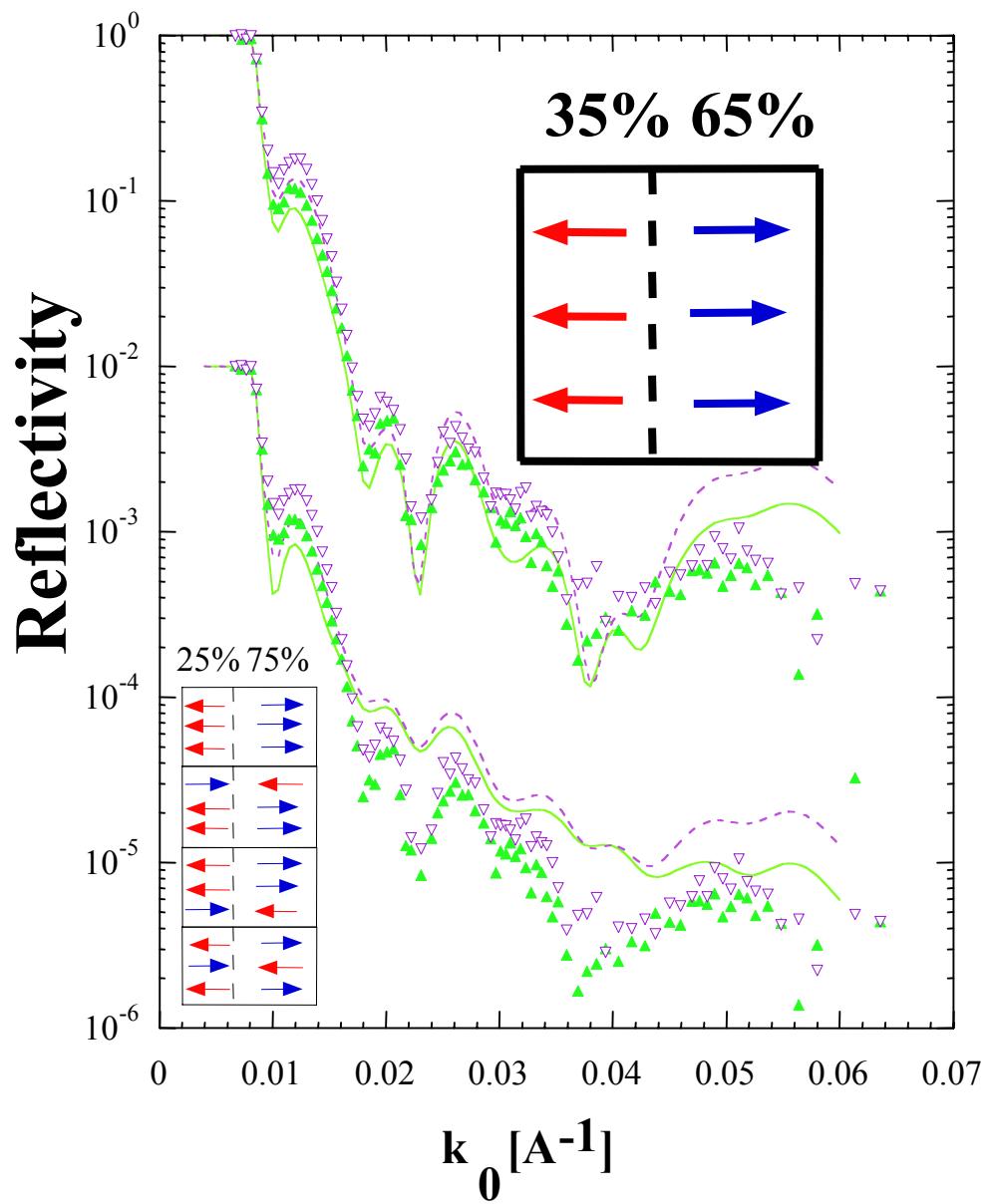


# Asymmetric reversal = largest exchange bias





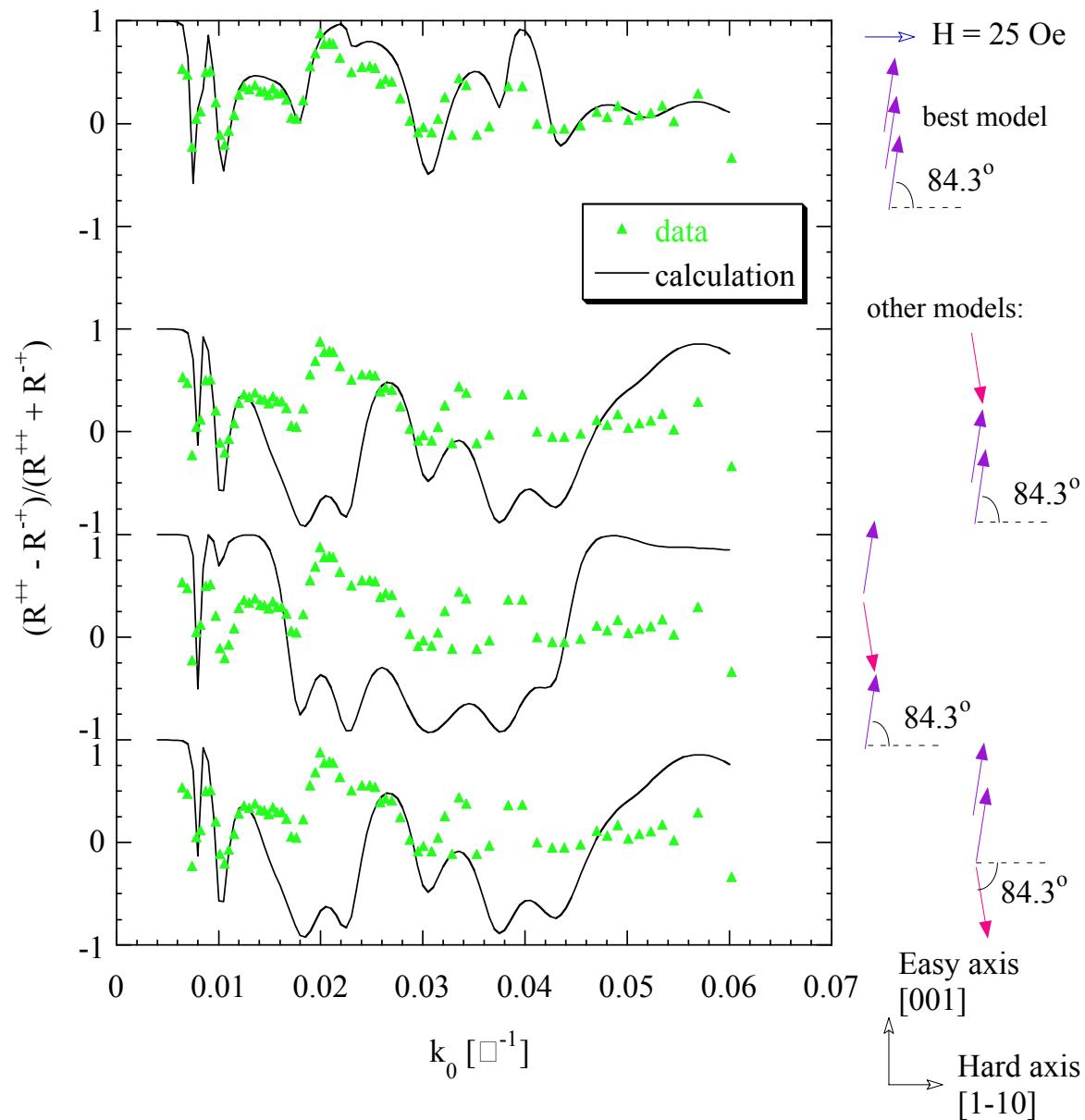
$t_{Cr} = 31 \text{ \AA}$ , H//easy axis



Only two types of domains with all three moments parallel

Indication of FM coupling

$t_{\text{Cr}} = 31 \text{ \AA}$ , H// hard axis

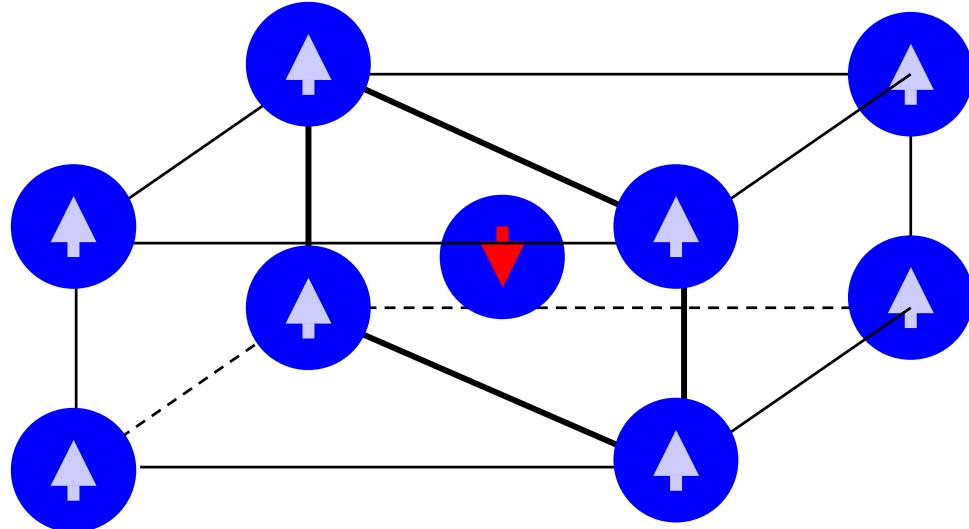


- Saturated along the hard axis
- Measured in small residual field of 25 Oe

All three moments are parallel

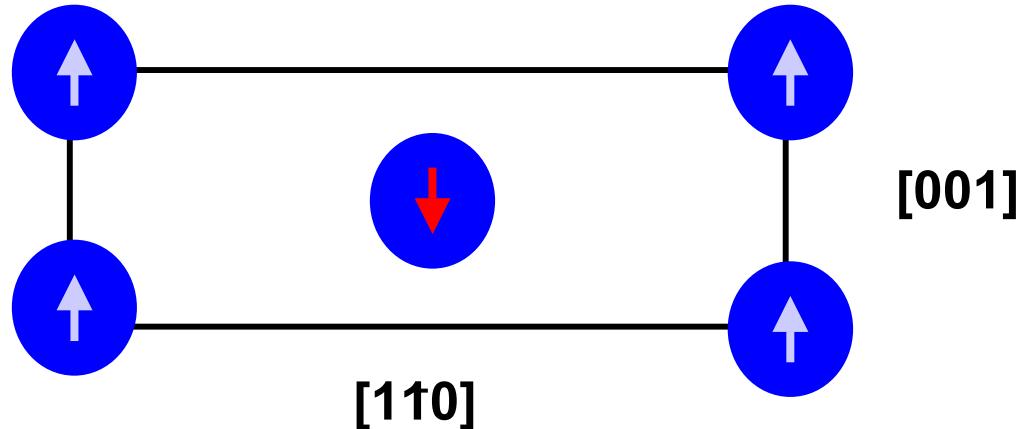
Indication of FM coupling

# $\text{FeF}_2$ , $\text{MnF}_2$ , $\text{CoF}_2$ , $\text{ZnF}_2$

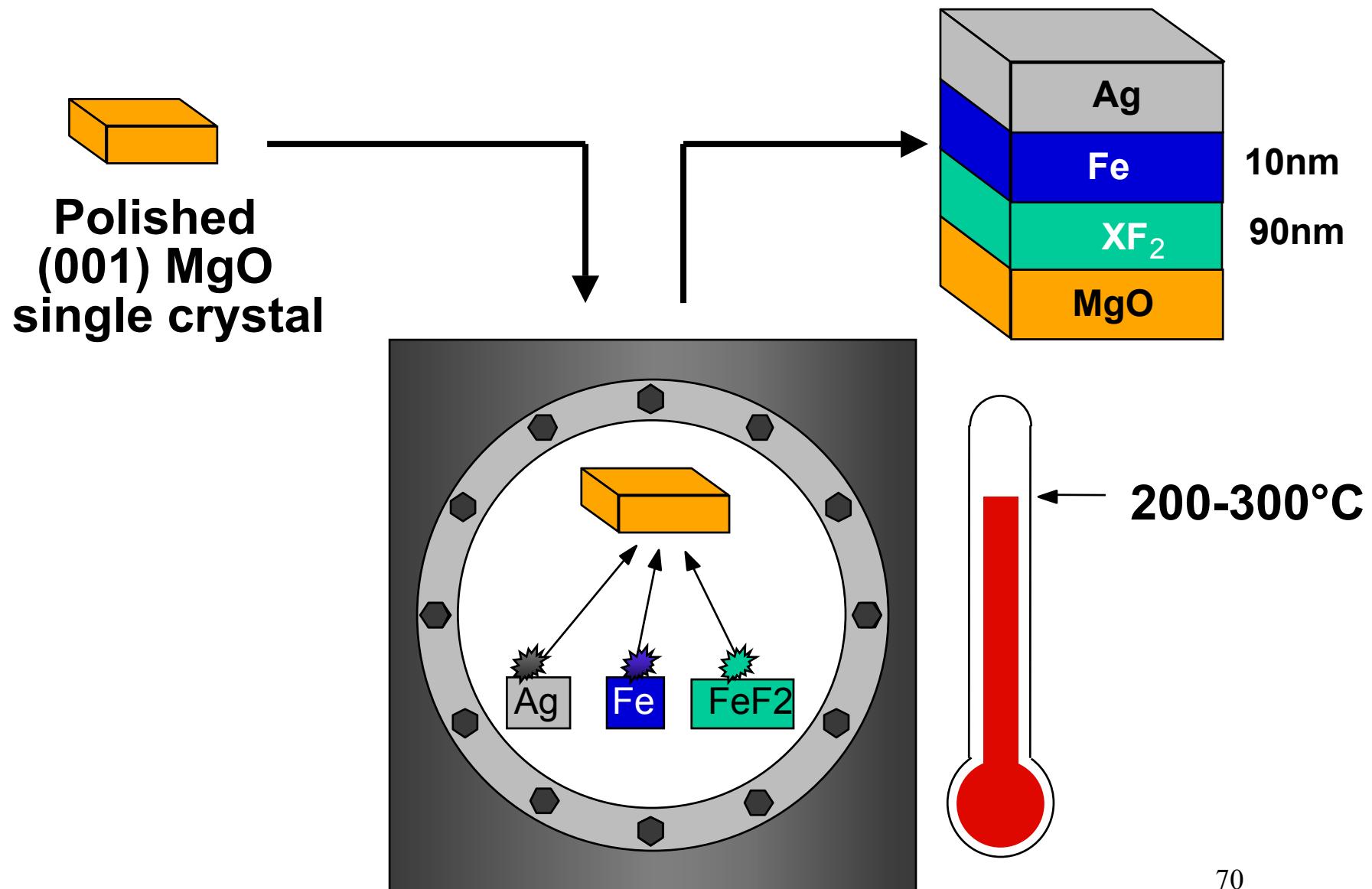


110 Interface

- Compensated AF surface.
- Simple spin structure.



# Fe- *twinned* $X\text{F}_2$



# Interlayer exchange coupling

The exchange coupling  $J$  between ferromagnetic layers across a non-ferromagnetic spacer is oscillatory.

- Ferromagnetic coupling
- Antiferromagnetic coupling
  - AFM alignment at zero field
  - Spin flop transition at high field
- Biquadratic coupling
  - 90° or non-collinear alignment
- Long period ( $\approx 18\text{\AA}$ , Fe/Cr)
- Short period ( $\approx 2\text{\AA}$ , Fe/Cr)
  - Only observed for very smooth interfaces

Review: M.D. Stiles JMMM 200 (1999) 322