Probing interfaces in magnetic nanolayers with soft x-ray standing waves

See-Hun Yang

Materials Sciences Division and Advanced Light Source
Lawrence Berkeley National Laboratory

- *Introduction to techniques*
  - *X-ray optics and electron/x-ray emission processes in spectroscopy*
  - *Depth-resolved information using a standing wave generator (SWG)*
- *Magnetic moments and magnetic order at buried interfaces in magnetic nanolayers: **Cr/Fe***
  - *Cr and Fe 3s high-resolution photoemission: local magnetic moment*
  - *X-ray magnetic circular dichroism (XMCD) in Cr and Fe 2p and 3p photoemission : magnetic order*
- *Conclusion and future work*

Berkeley Spectroscopy Club , 09/26/01

Why study interfaces in magnetic multilayers ?

- spin dependent scattering of electrons at interface
magnetic anisotropy, exchange bias near interface
® GMR, MTJ, TMR, spin valve,…

- chemical & magnetic variation at interface

Fe/Cr : giant MR (Fe: F, Cr: AF)
MR > 40 % at T=4.5 K and 10 % at T=300 K

Some questions :
- *degree of interdiffusion at interfaces*
- *chemical and magnetic variation or roughnesses*
- *distribution of magnetic order and magnetic moment near interface (A. Fujimori et al; C. Turtur et al.)*

to be discussed here *ongoing or future project*

Specific projects (Berkeley, Oct. 1998 – present)

-*general consideration of full x-ray optics and electron emission processes in spectroscopy*

® *first-of-its-kind C program, all effects included > 5K lines (to be submitted to Phys. Rev. B)*

-*novel uses of multilayer standing wave generators (SWG) in spectroscopy*

® *depth-resolved photoemission (Surf. Sci. Lett, 461 L557 (2000))*

® *non-destructive probing buried interface in magnetic nanolayer (to be submitted to Science)*

- *multi-atom resonant photoemission (MARPE)*

® *detailed analysis of macroscopic x-ray optical view of MARPE (Phys. Rev. B 63, 115119 (2001); to be submitted to Phys. Rev. Lett.)*

Semi-infinite layer : bulk & surface

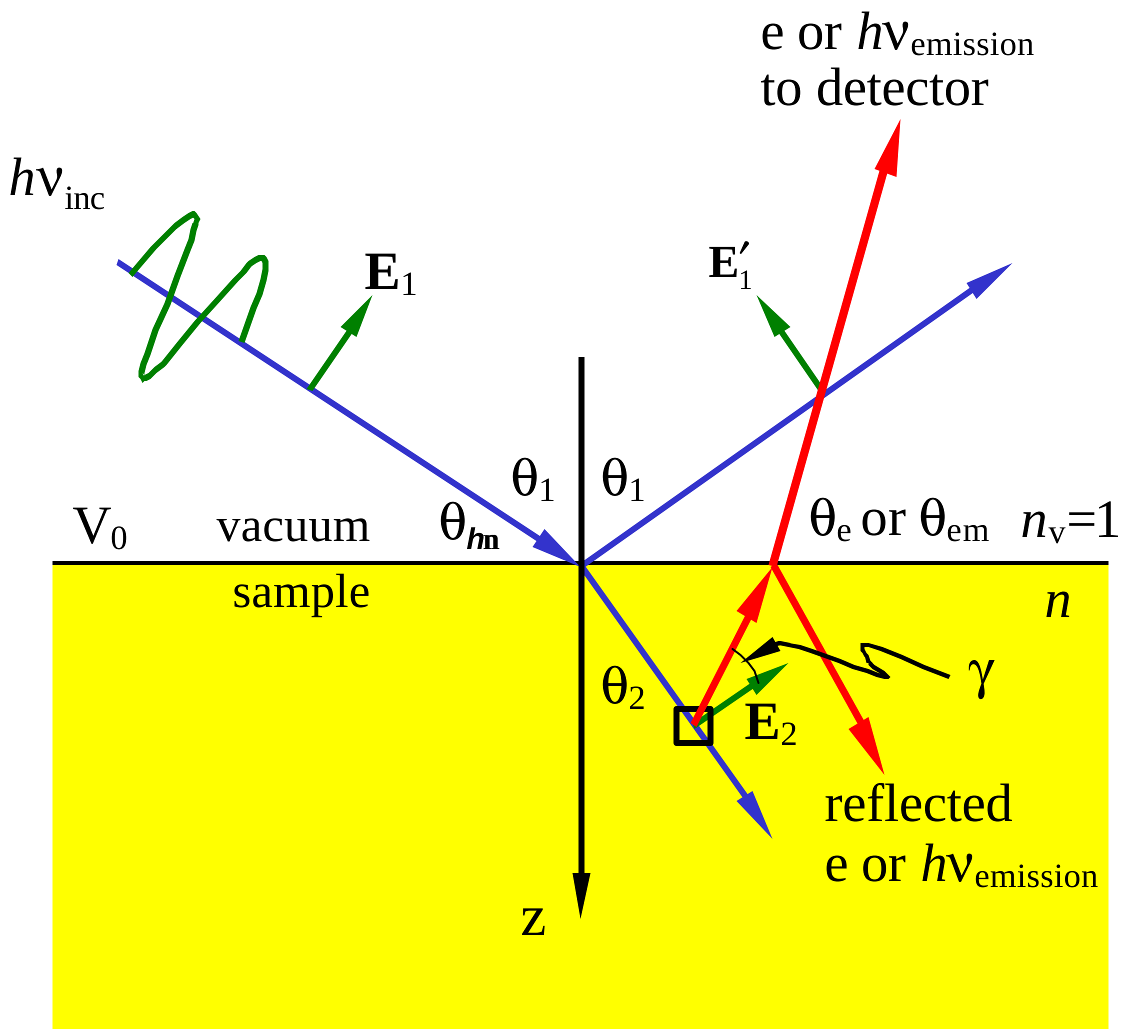
X-ray Optics : hn_{inc}

- *optical constant*
  $n\,(hn) = 1 - d\,(hn) + i\,b(hn)$
  (Henke, Gullikson, Davis)
  scattering form factors f_1, f_2
- *absorption, transmission*
  (Henke et al., Windt)
- *variable x-ray polarization*
  (s, p, RCP, LCP and mixed)

Photoelectron emission : e

- *atomic cross section (Yeh, Lindau)*
- *asymmetry factor (Fadley)*
- *electron inelastic attenuation*
  (Tanuma, et al)
- *reflection & refraction of*
  electron at surface (Fadley)

X-ray emission : $hn_{emission}$



Multilayer : Standing Wave Generator

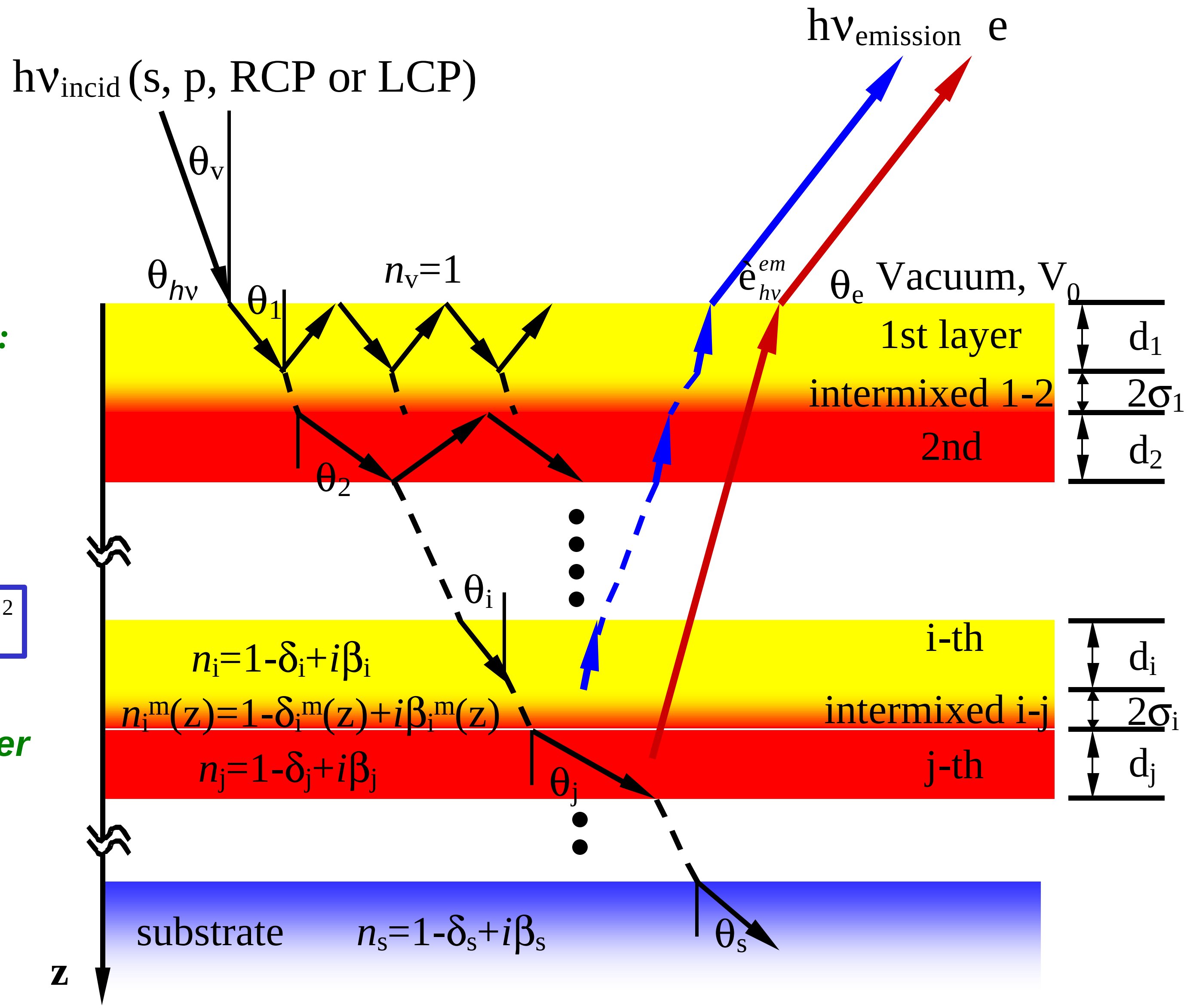
Calculations :

- multiple reflection & refraction

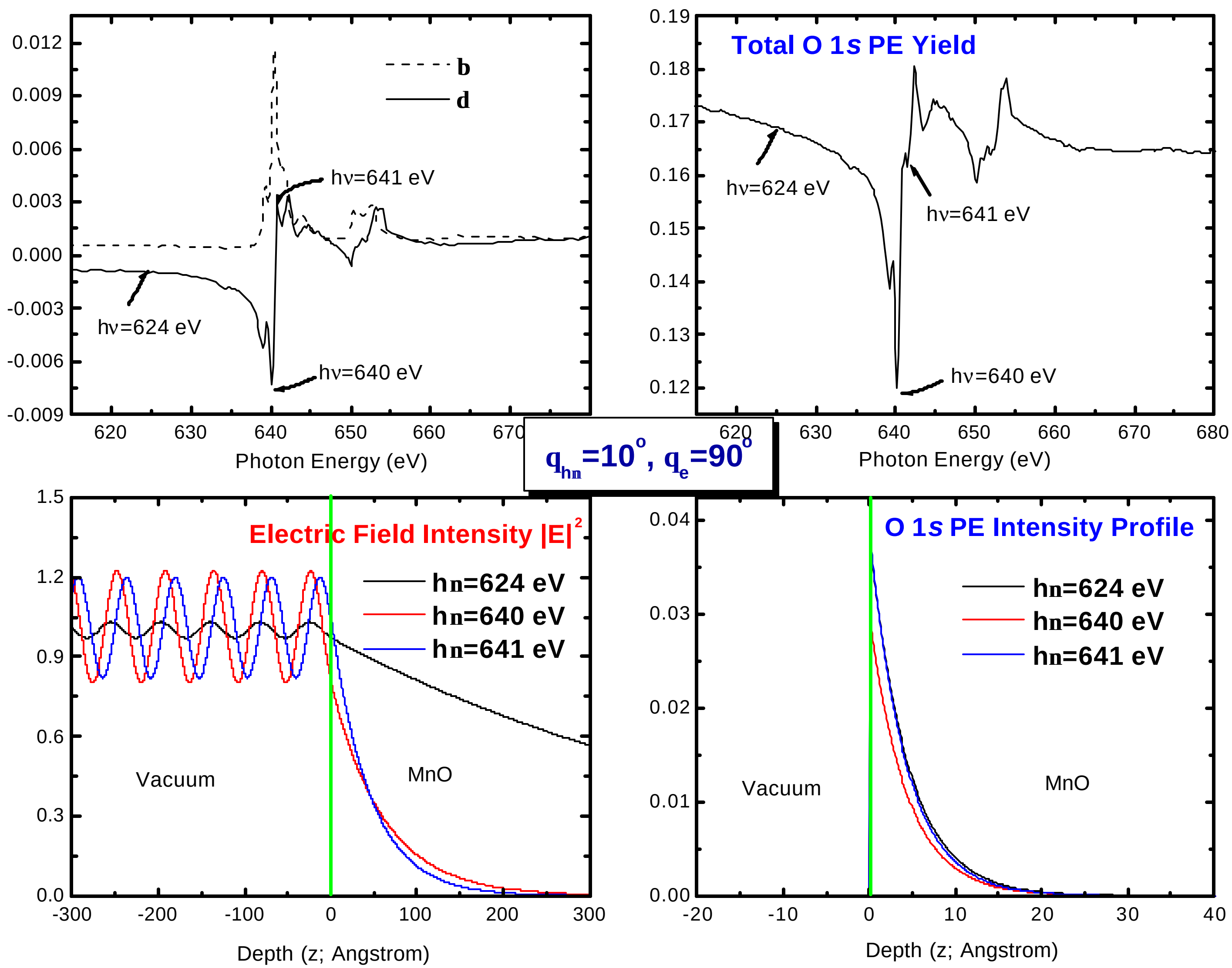
- intermixing ($s_i(z)$) : exact numerical treatment of interface diffusion profile

$$I_i(z) = |\mathbf{E}_i^+(z) + \mathbf{E}_i^-(z)|^2$$

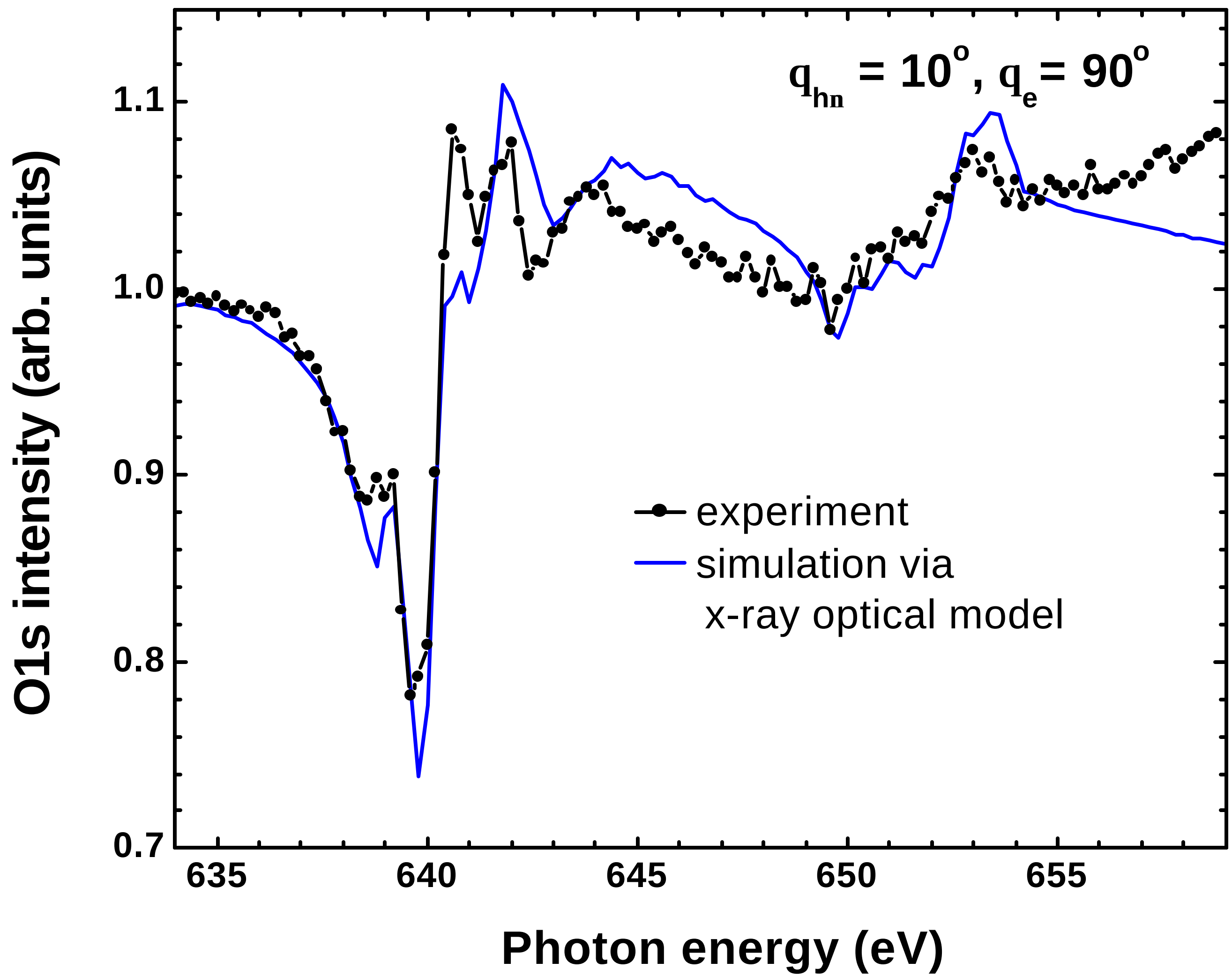
= electric field intensity at i-th layer (cf. Windt)



A simple test : *O 1s photoemission yield from semi-infinite MnO around Mn 2p edges*

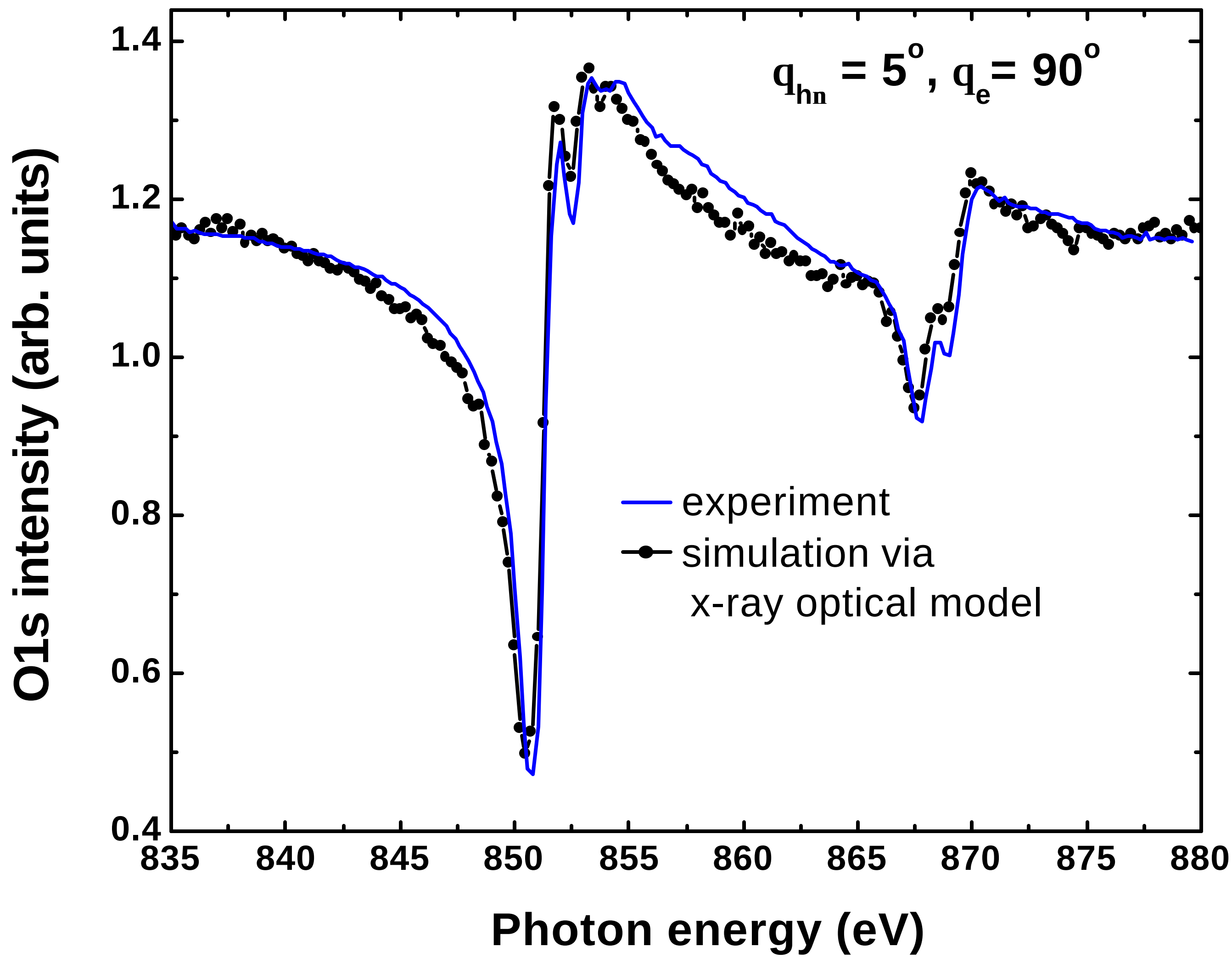


O 1s modulation around Mn 2p edge in MnO(001)



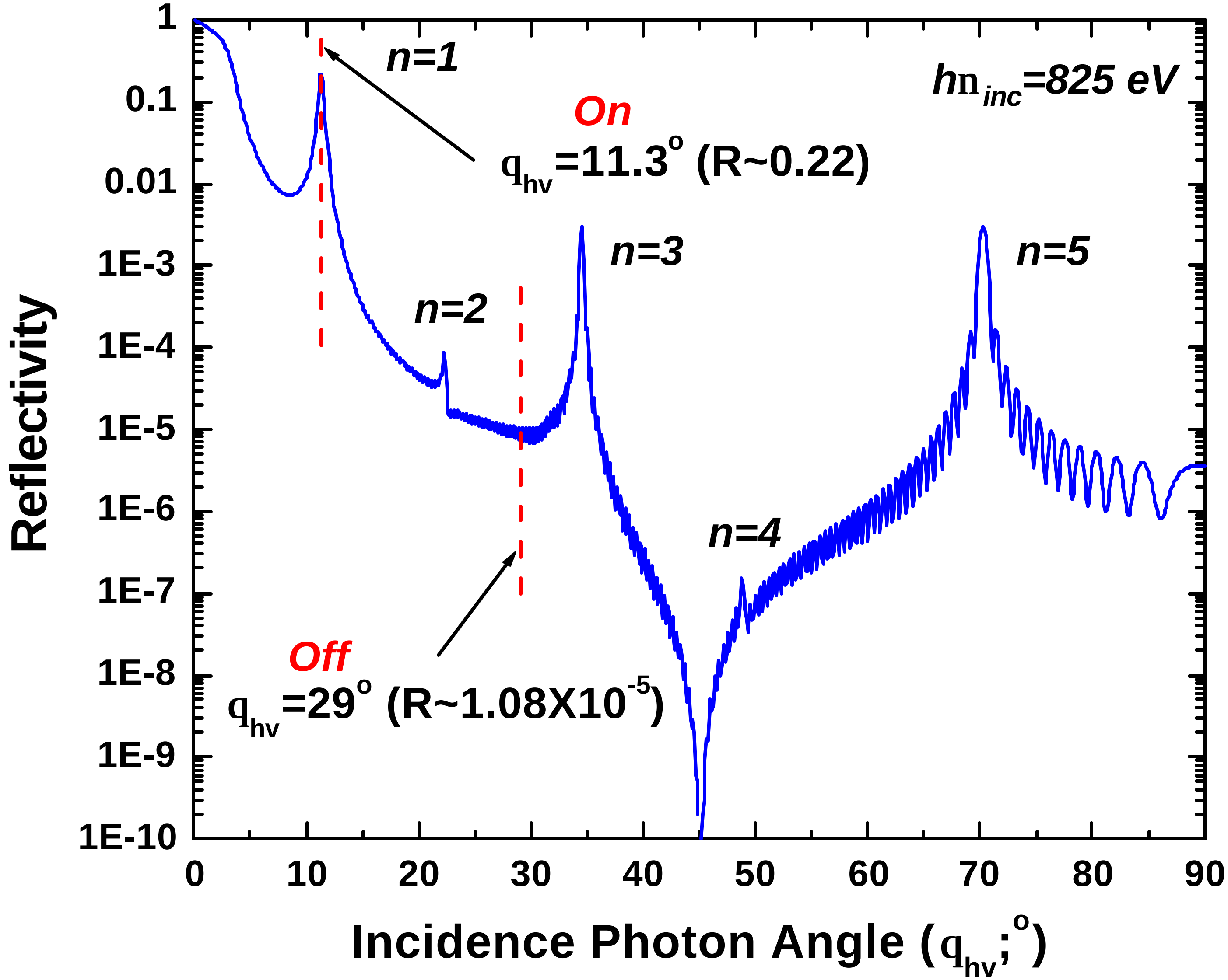
A. W. Kay, F. J. Garcia de Abajo, S.-H. Yang, et al
Phys. Rev. B 63 115119 (2001)

O 1s modulation around Ni 2p edge in NiO(001)



N. Mannella, S.-H. Yang, L. Zhao, B. S. Mun, et al.
to be submitted to Phys. Rev. Lett.

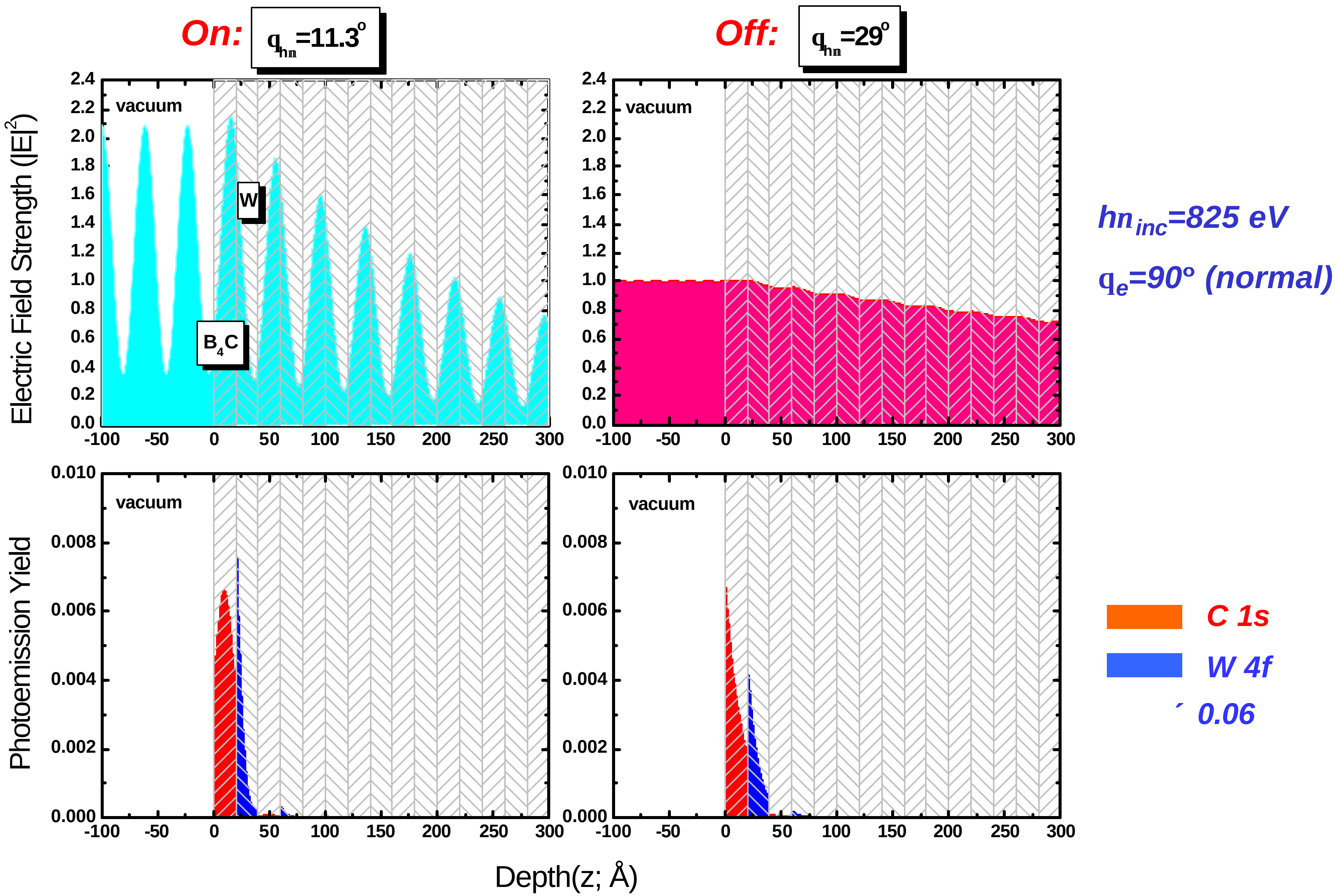
Multilayer reflectivity : $[B_4C\ (20\ \text{Å})\ /W\ (20\ \text{Å})]_{20}/Si$



Bragg condition

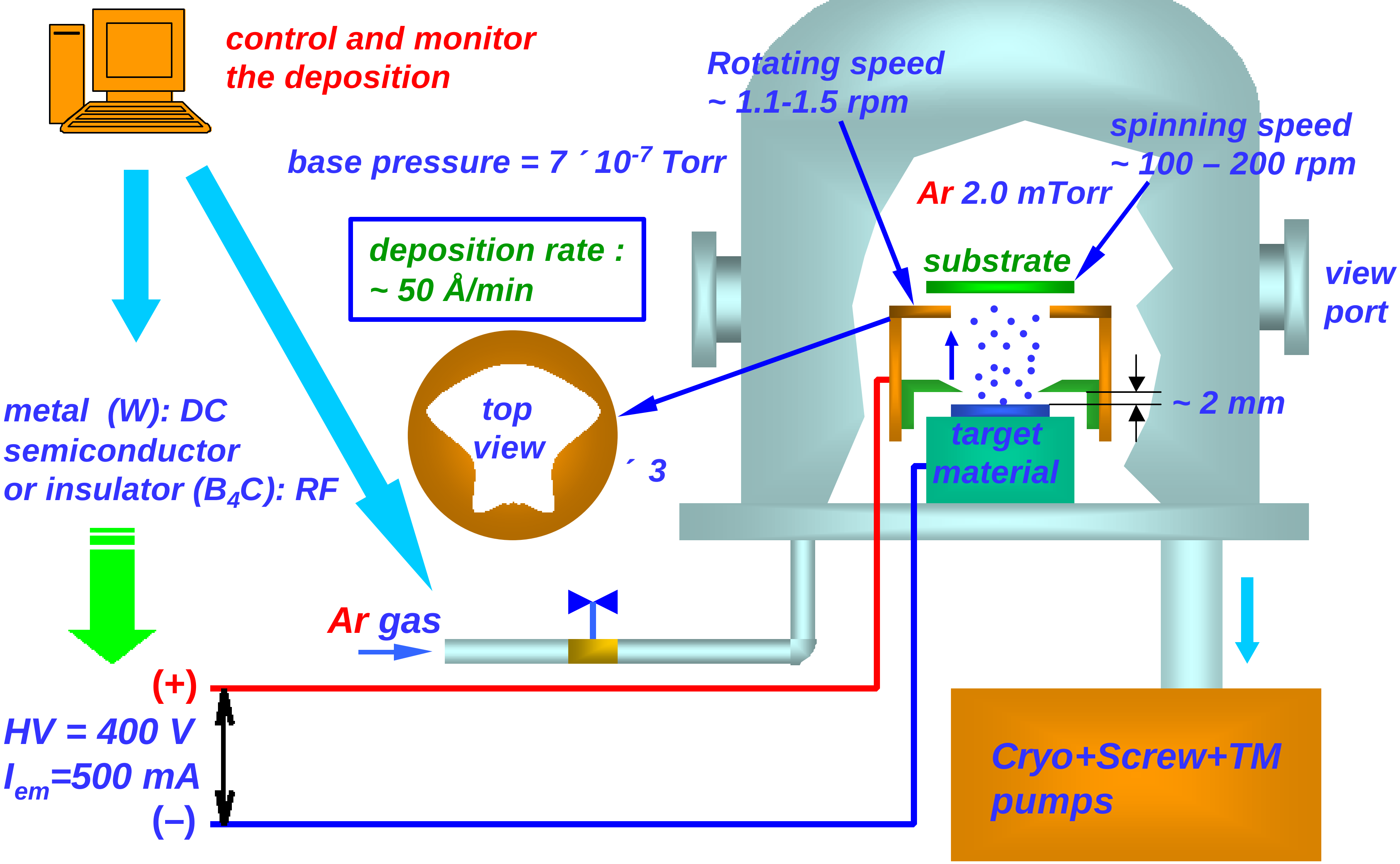
$n\lambda = 2d \sin\theta_{h\nu}$

$[B_4C\ (20\ Å)\ /W\ (20\ Å)]_{20}/Si$

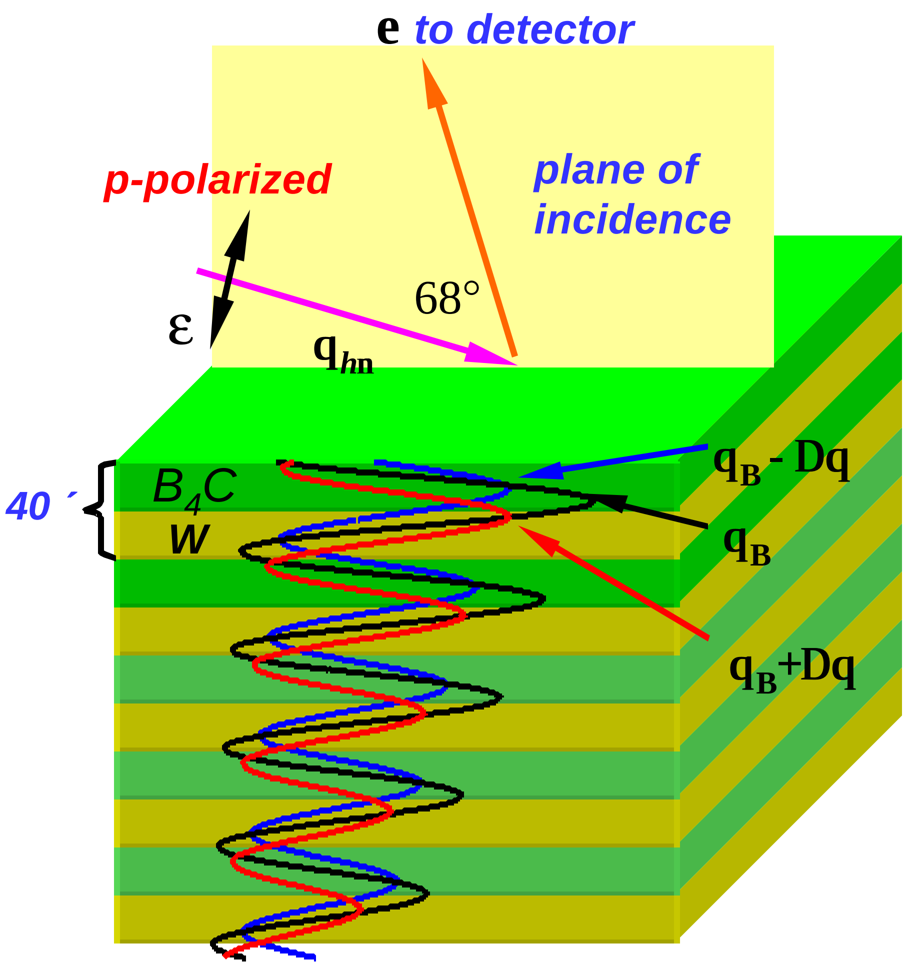


Sputter Chamber for Multilayer Growth

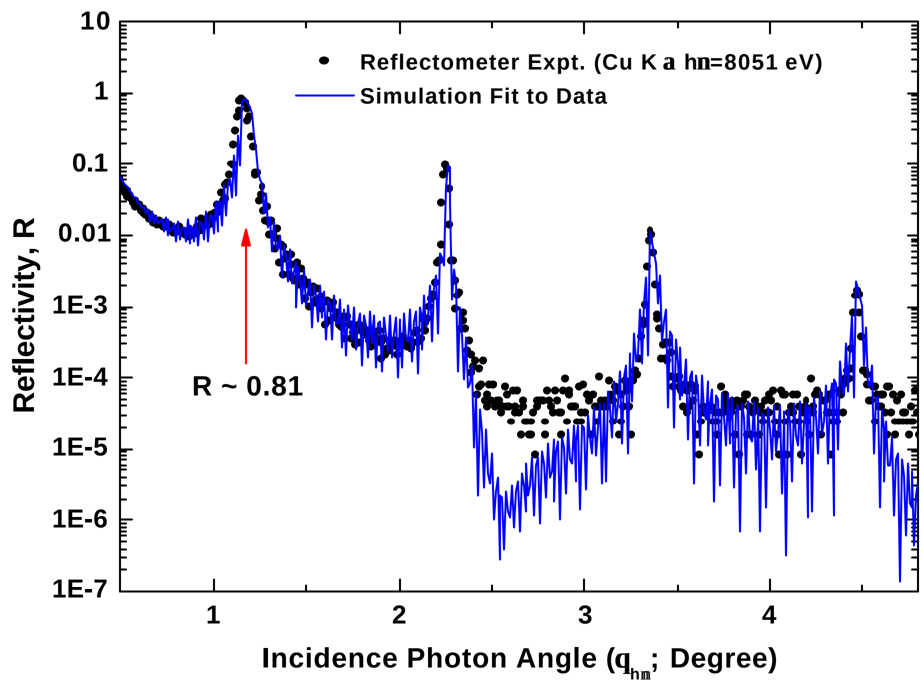
Center for X-ray Optics (CXRO):
J. Kortright, J. Underwood



Multilayer Soft X-ray Standing Wave Generator (SWG)



Hard x-ray reflectivity : ~ 8.0 keV



- *light bake in situ (~70 ºC), no other cleaning*
- *period = 39.6 Å, $[B_4C(22.5\ Å)/W(17.1\ Å)]_{40}$ on SiO_2/polished Si(111)*
- *interface diffusion length s = 2.4 Å*

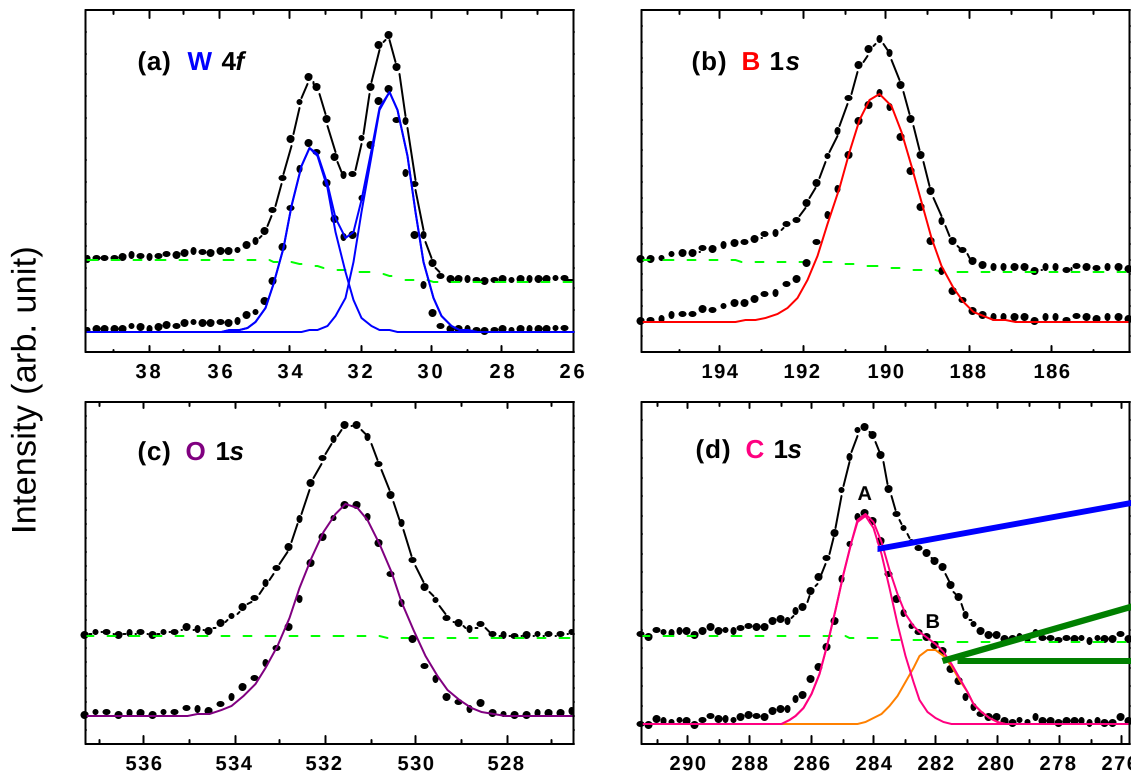
Previous work on photoemission from a SWG

core XPS from $[Mo/B_4C]_{30}$, Hayashi et al., (Appl. Phys. Lett. 68, 1921 (1996))

~2.714 keV, unable to resolve unique behavior of different Mo, B and C peaks

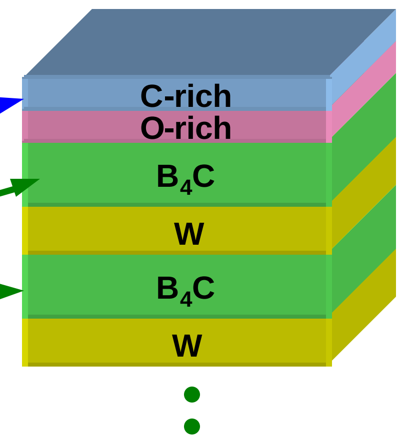
Our work

***hν*=750 eV, *θ*$_{h\nu}$=7.85 °**



Binding Energy (eV)

- *bend magnet beamline 9.3.2 at the Advanced Light Source*
- *x-ray beam divergence of 1.2 mrad vertical x 2.4 mrad horizontal*



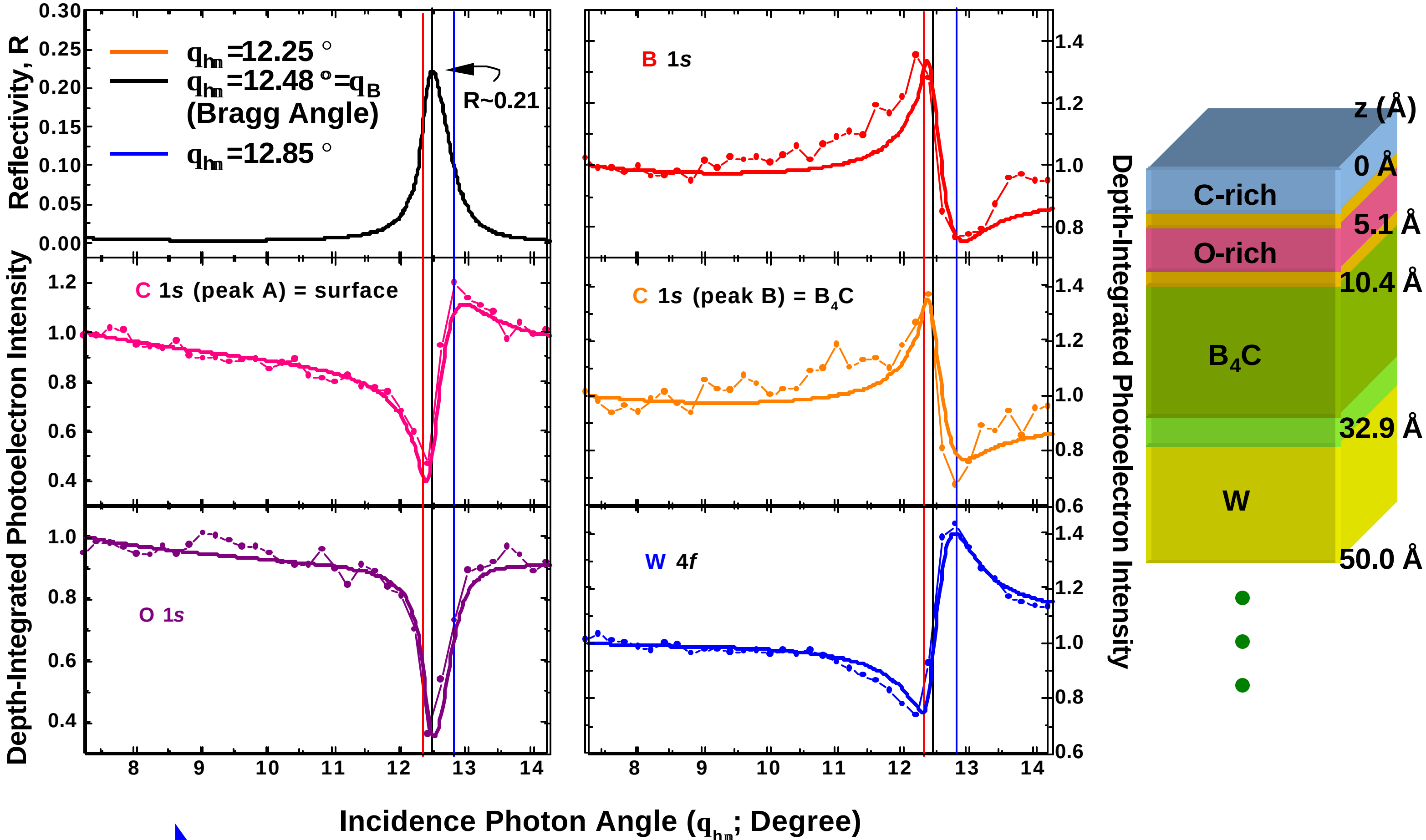
A=surface layer

$B=B_4C$

S.-H. Yang et al.
Surf. Sci. Lett, 461 L557 (2000))

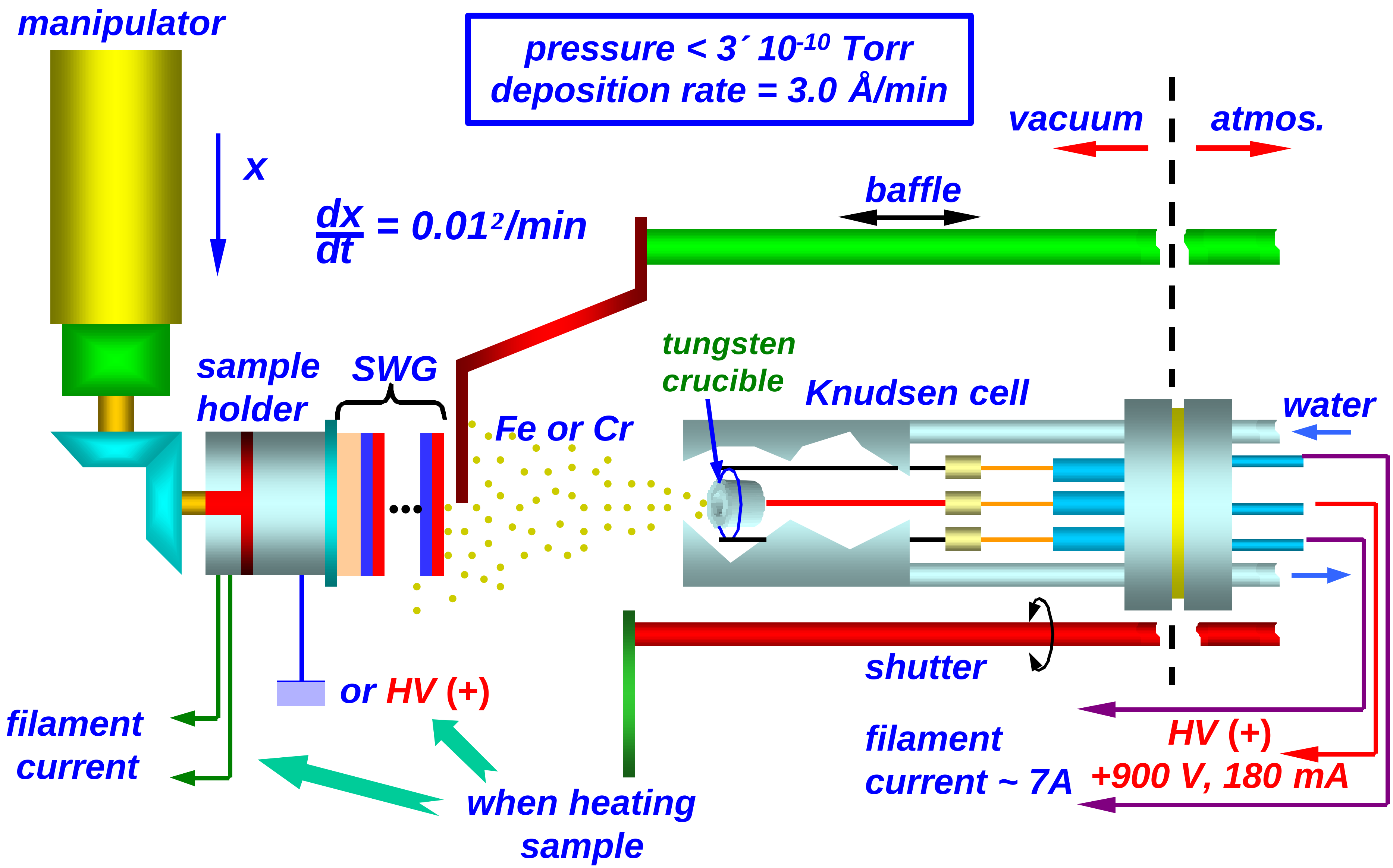
“Rocking curves” of photoemission yield for each core level

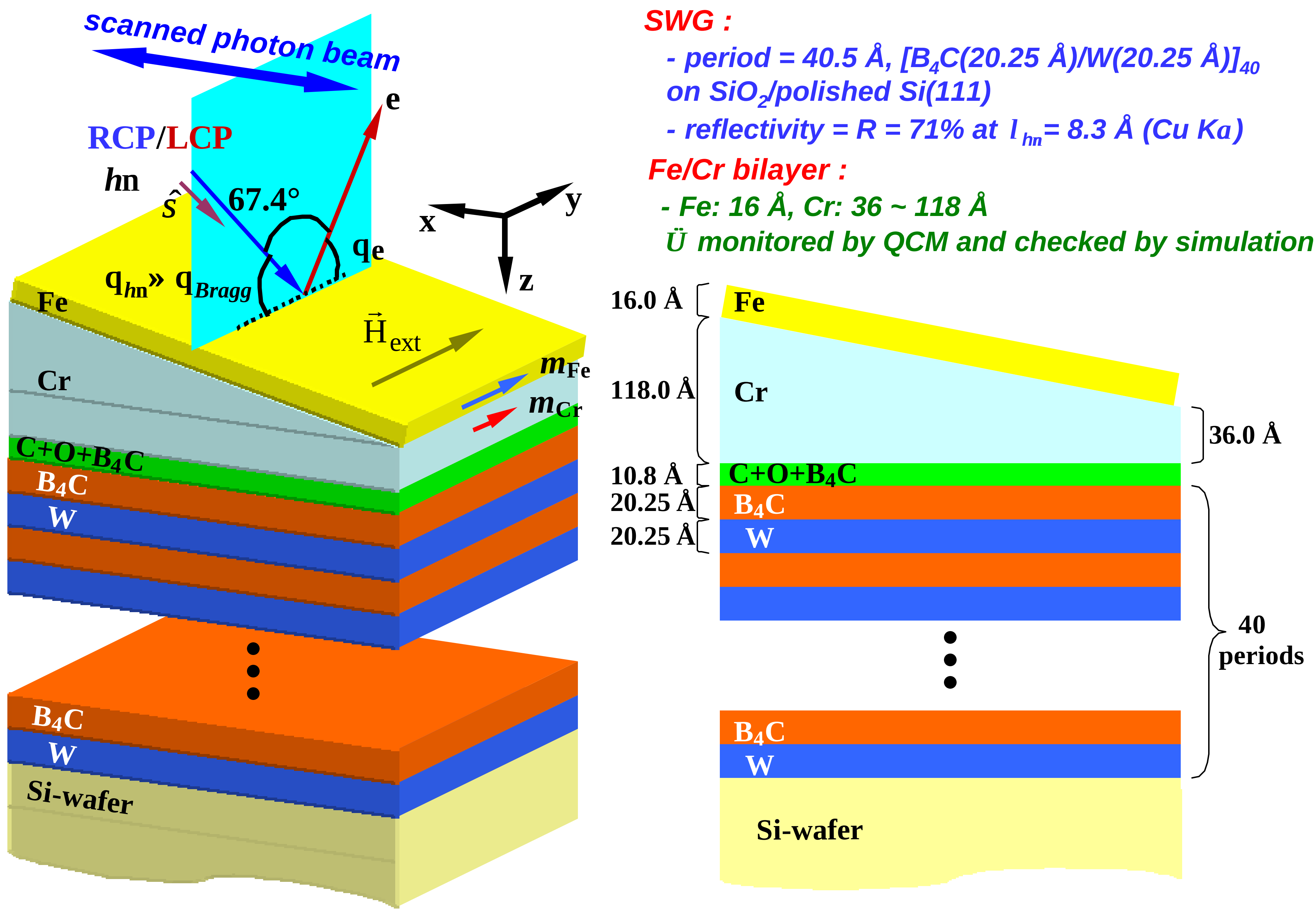
***Experiment & Calculation** → C-rich(5.1 Å)/O-rich(5.3 Å)/[B_4C(22.5 Å)/W(17.1 Å)]$_{40}$/SiO_2*



→ *Excellent Agreement between Expt & Calc*

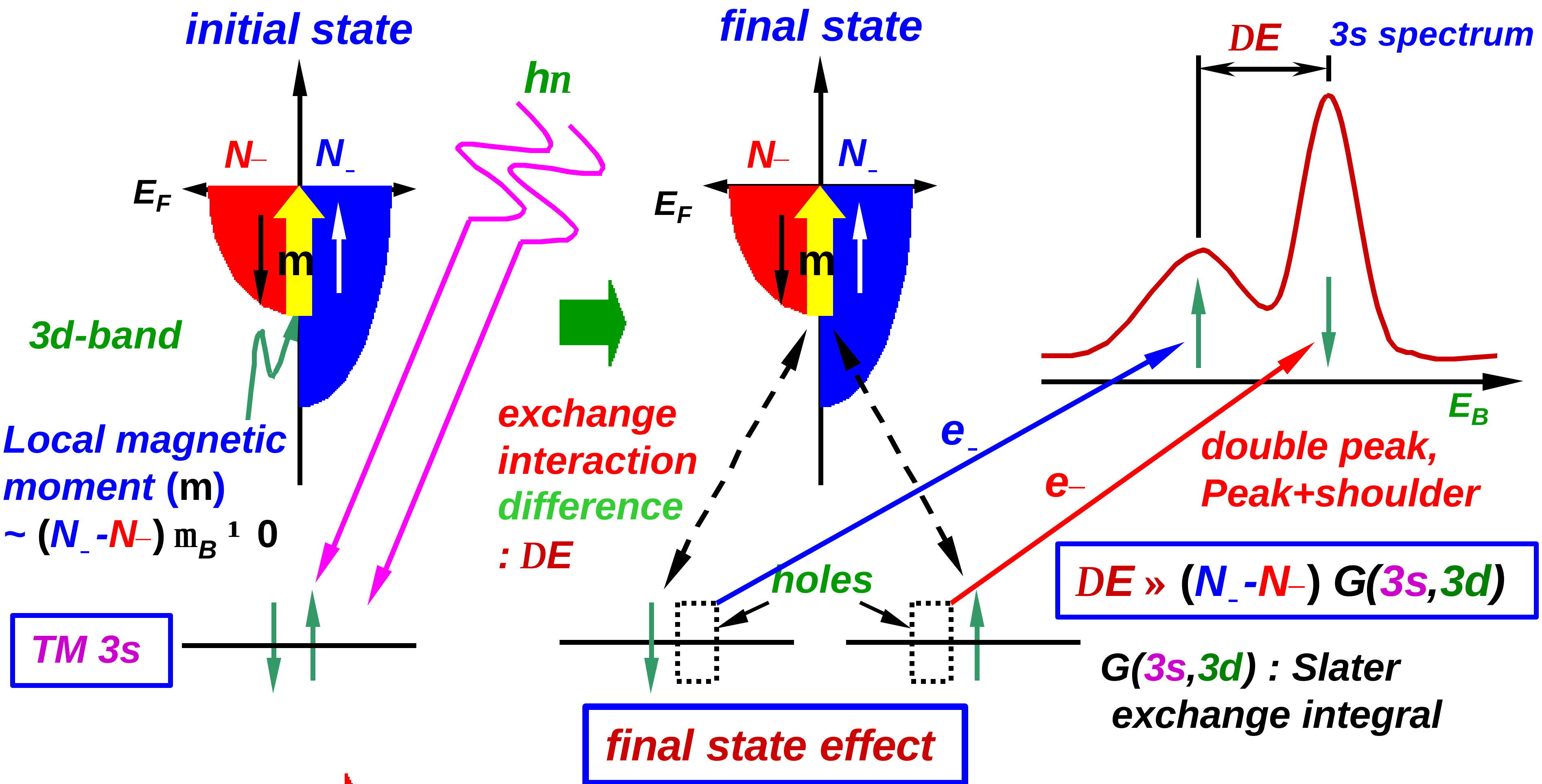
In-situ MBE growth of Fe/Cr (wedge) bilayer on SWG





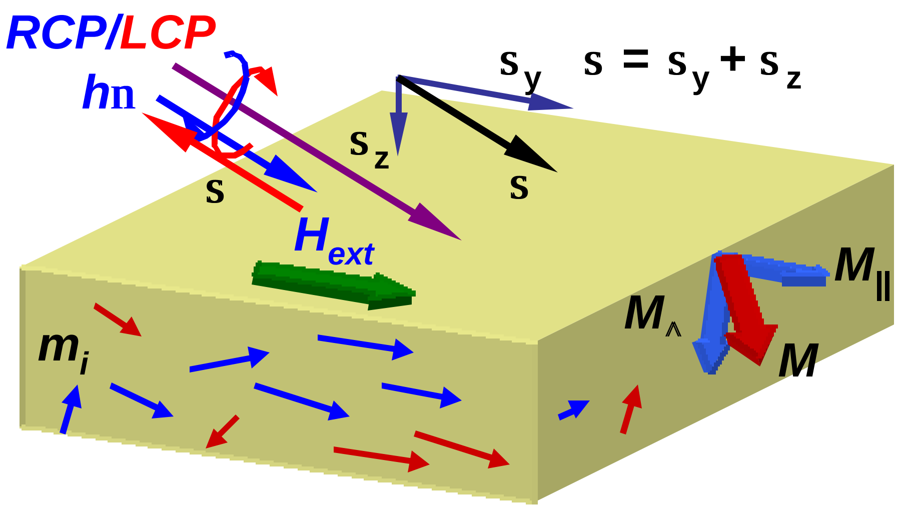
scanned photon beam
RCP/LCP
hn
ŝ
67.4°
e
q_e
q_hn» q_Bragg
x
y
z
H_ext
m_Fe
m_Cr
Fe
Cr
C+O+B4C
B4C
W
B4C
W
Si-wafer
SWG :
- period = 40.5 Å, [B4C(20.25 Å)/W(20.25 Å)]40
on SiO2/polished Si(111)
- reflectivity = R = 71% at l hn= 8.3 Å (Cu Ka)
Fe/Cr bilayer :
- Fe: 16 Å, Cr: 36 ~ 118 Å
Ü monitored by QCM and checked by simulation
16.0 Å
118.0 Å
10.8 Å
20.25 Å
20.25 Å
Fe
Cr
36.0 Å
C+O+B4C
B4C
W
40
periods
B4C
W
Si-wafer

3s spectrum for a magnetic 3d-transition metal



3s splitting DE : measure of the magnitude of local magnetic moment

Magnetic Circular Dichroism (MCD) in Photoemission (PE)



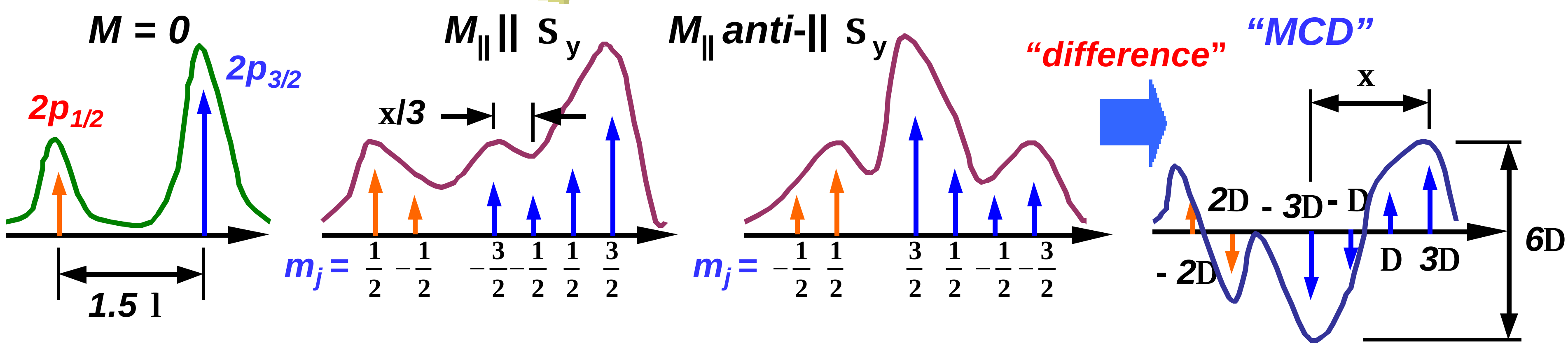
$M = S_i\, m_i = M_{||} + M_{\wedge}$

$M_{||} = M_y$

$M_{\wedge} = M_x + M_z = 0$ (\ H_{ext} || y)

Hamiltonian

$H = l\, l \bullet s + x S_y$



MCD strength µ $s \bullet S_i\, m_i = s_y M_{||}$

x or 6D

D µ x

MCD in PE : measure of ferromagnetic order along y-direction

Why MCD in photoemission and not in absorption?
Why is WEDGE crucial ?

- ***Problems in X-ray absorption MCD of SWG***

  A. optical properties are altered when photon energy is swept
  B. SW's altered when incidence angle is scanned

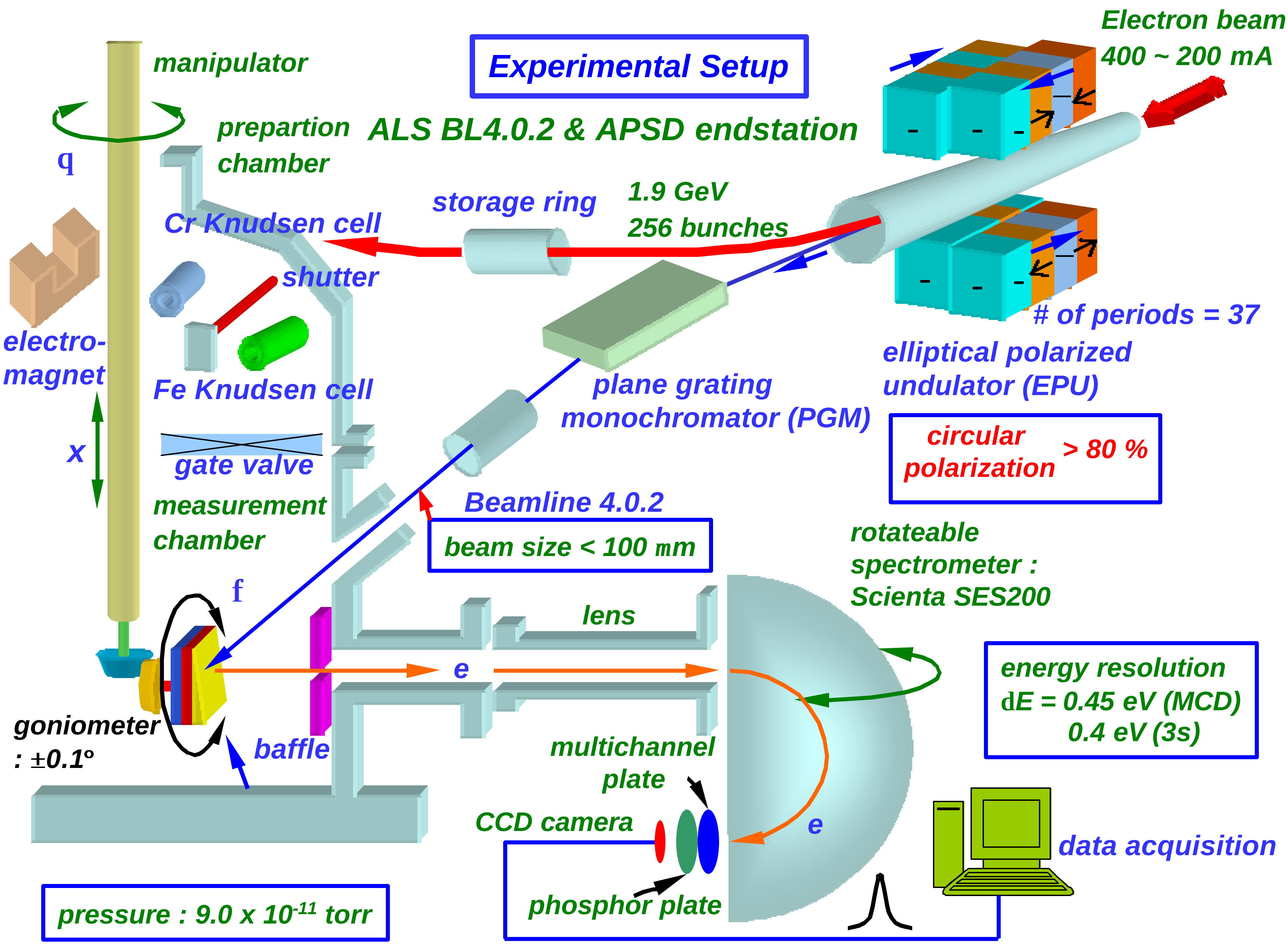
  { ***Bragg angles change***
  Phases of SW change
  Contrasts of SWG change } ***as a function of photon energy***

  ➡ ***Too many free parameters to be determined --- see e. g.***
  S. K. Kim and J. Kortright (LBNL), Phys. Rev. Lett, 86, 1347 (2001)

- ***Our study***

  - photoemission MCD solves problem A
  ⬅ ***do not need to scan the photon energy; element-specific***
  - wedge solves problems A and B
  ⬅ ***do not need to scan the incidence angle; phase pinning***

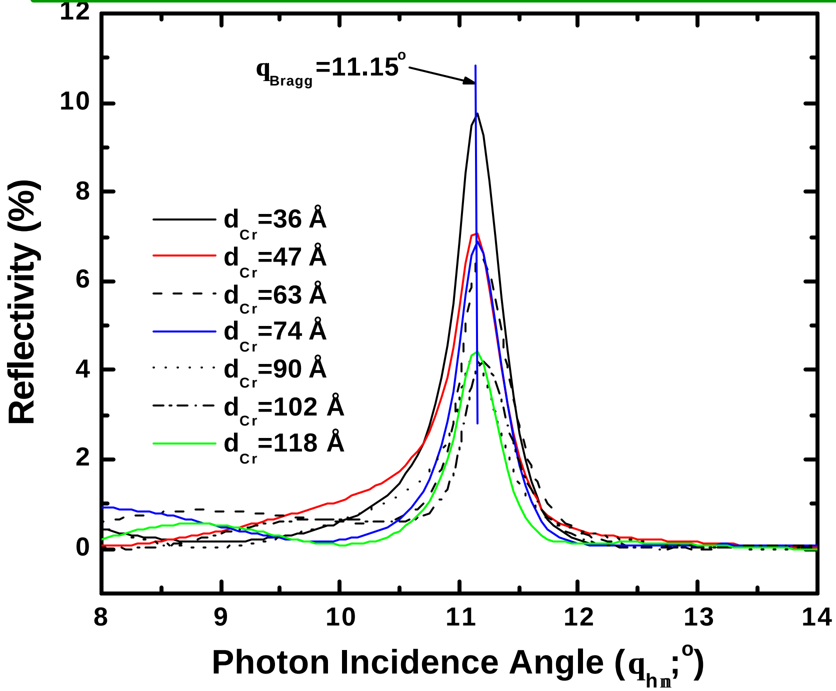
➡ ***This work : minimizes # of free parameters***
improves depth-resolution of SW

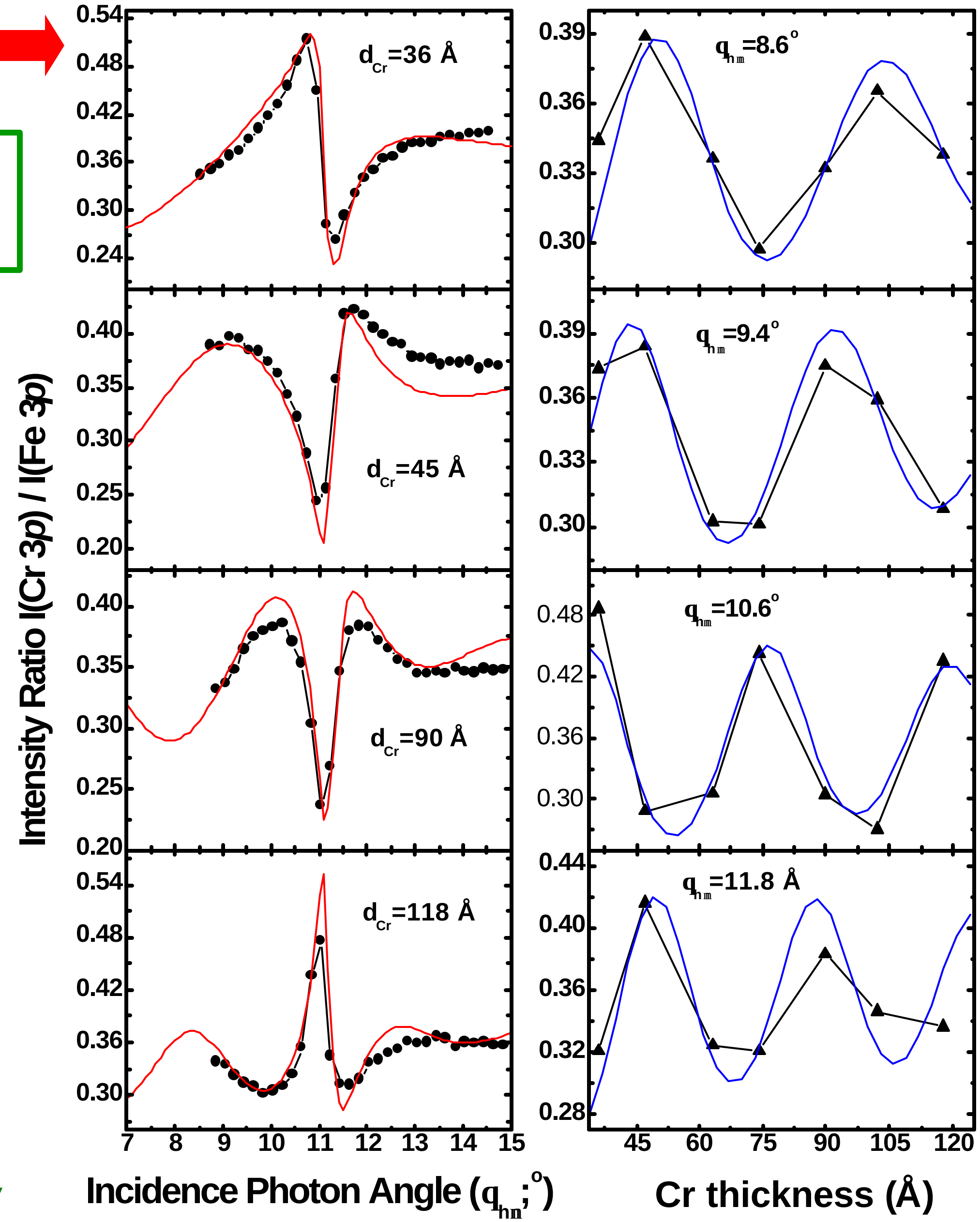

Experimental Setup
ALS BL4.0.2 & APSD endstation
manipulator
q
preparation chamber
Cr Knudsen cell
shutter
Fe Knudsen cell
electro-magnet
x
gate valve
measurement chamber
storage ring
1.9 GeV
256 bunches
Electron beam
400 ~ 200 mA
# of periods = 37
elliptical polarized undulator (EPU)
plane grating monochromator (PGM)
circular polarization > 80 %
Beamline 4.0.2
beam size < 100 mm
f
lens
e
rotateable spectrometer : Scienta SES200
energy resolution
dE = 0.45 eV (MCD)
0.4 eV (3s)
goniometer : ±0.1°
baffle
multichannel plate
CCD camera
phosphor plate
data acquisition
pressure : 9.0 x 10-11 torr

***Question** : How much are the standing waves affected by the bilayer Fe/Cr ?*

***Answer** : very little (< 0.2 Å shift)* *experimental + calculated photoemission yield ratio I(Fe 3p)/I(Cr 3p) in (Fe/Cr)+SWG*

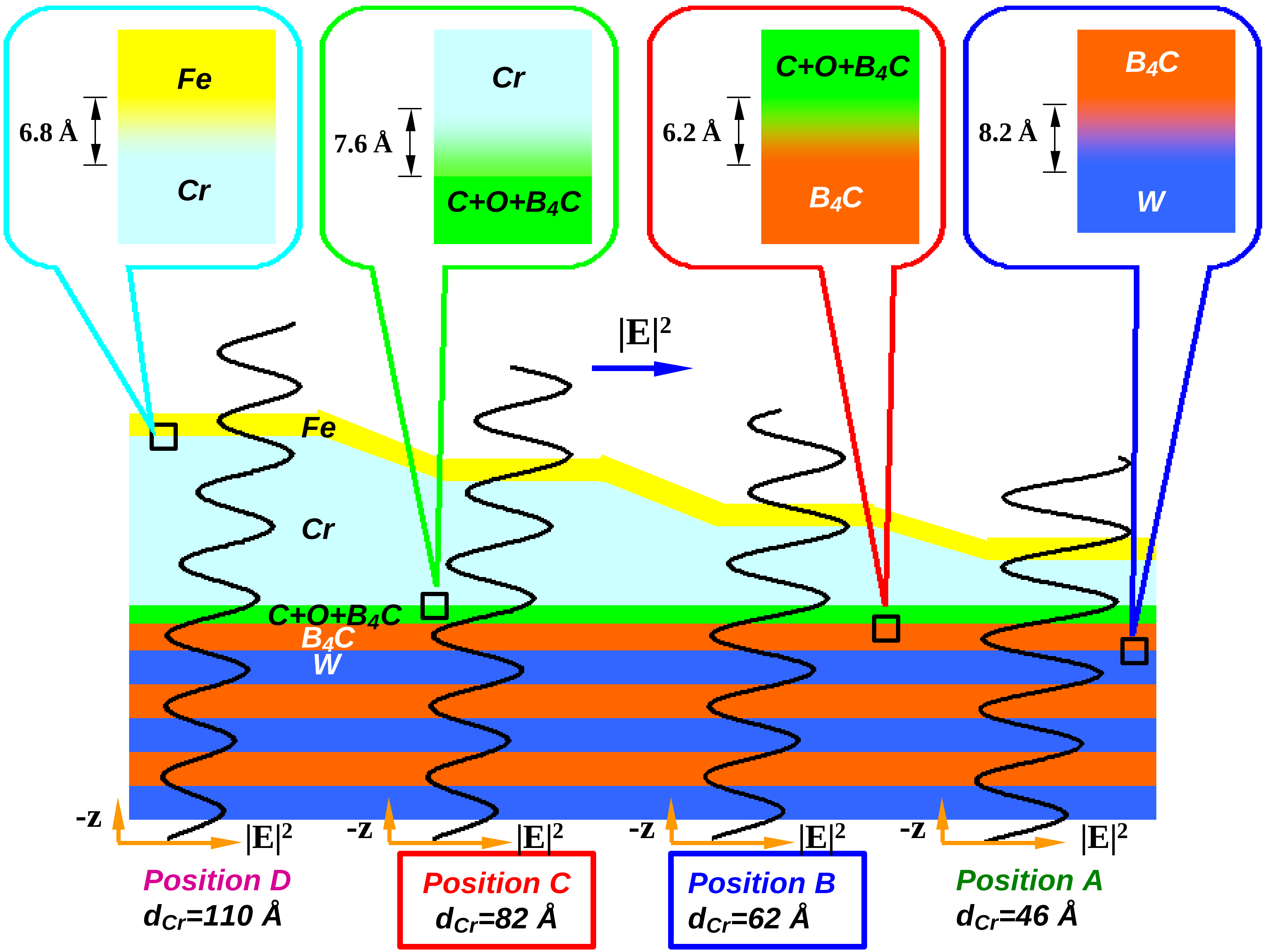
calculated reflectivity for wedge (Fe/Cr)+SWG





calculation of q_{Bragg}
® no shift with d_{Cr}

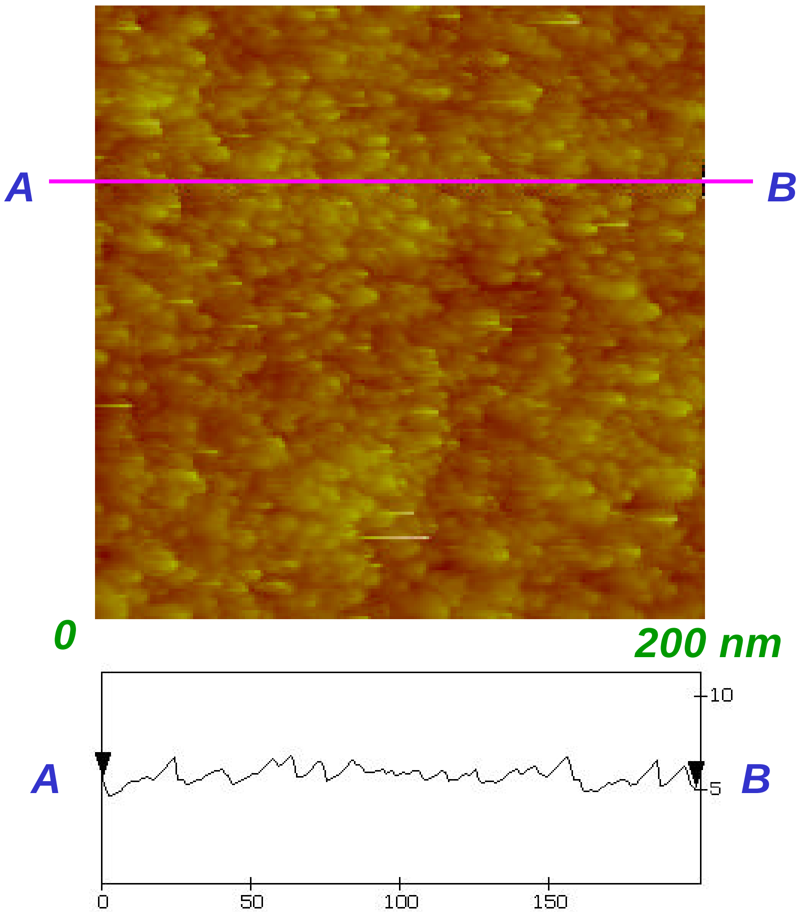
"phase pinning" of SWG vs. d_{Cr}



Fe
6.8 Å
Cr
Cr
7.6 Å
C+O+B$_4$C
C+O+B$_4$C
6.2 Å
B$_4$C
B$_4$C
8.2 Å
W
|E|^2
Fe
Cr
C+O+B$_4$C
B$_4$C
W
-z
|E|^2
Position D
d$_{Cr}$=110 Å
Position C
d$_{Cr}$=82 Å
Position B
d$_{Cr}$=62 Å
Position A
d$_{Cr}$=46 Å

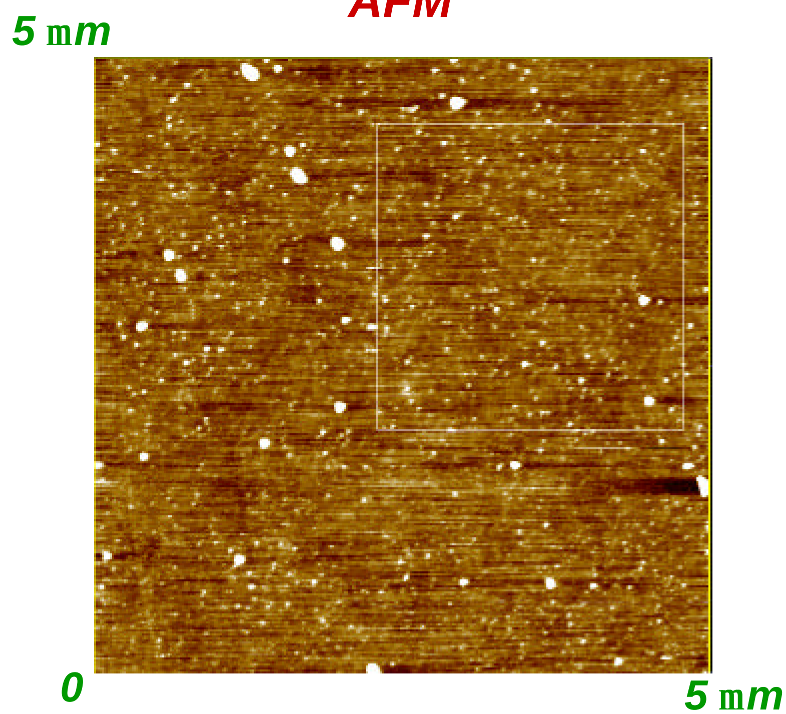
Scanning Probe Microscopy Image for Multilayer SWG

STM



average roughness = 2.8 Å

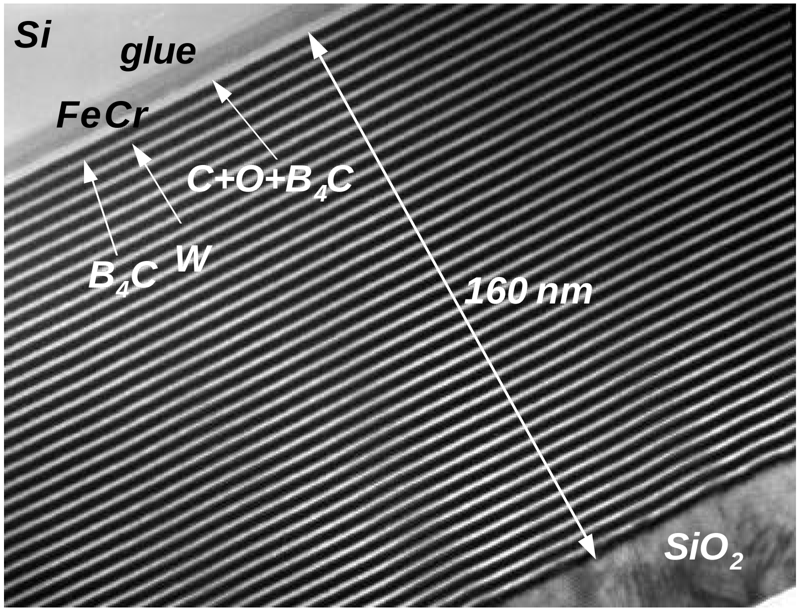
AFM

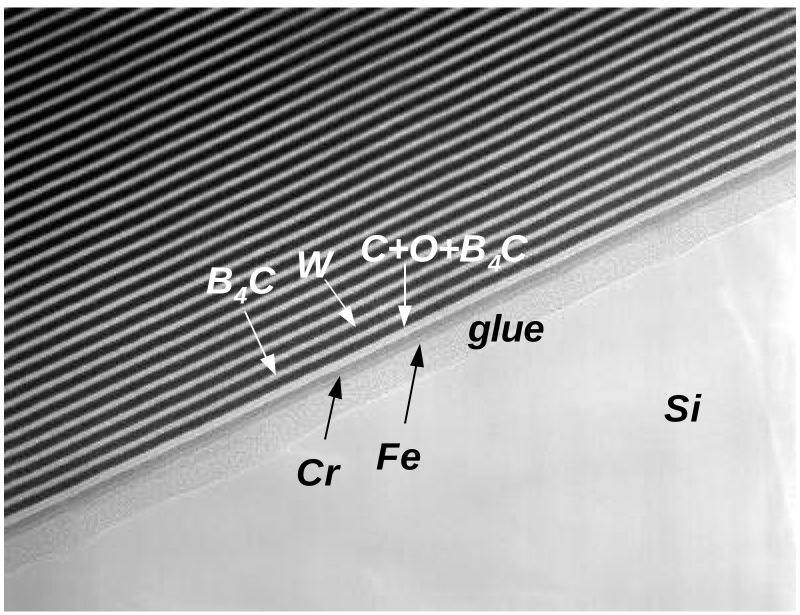


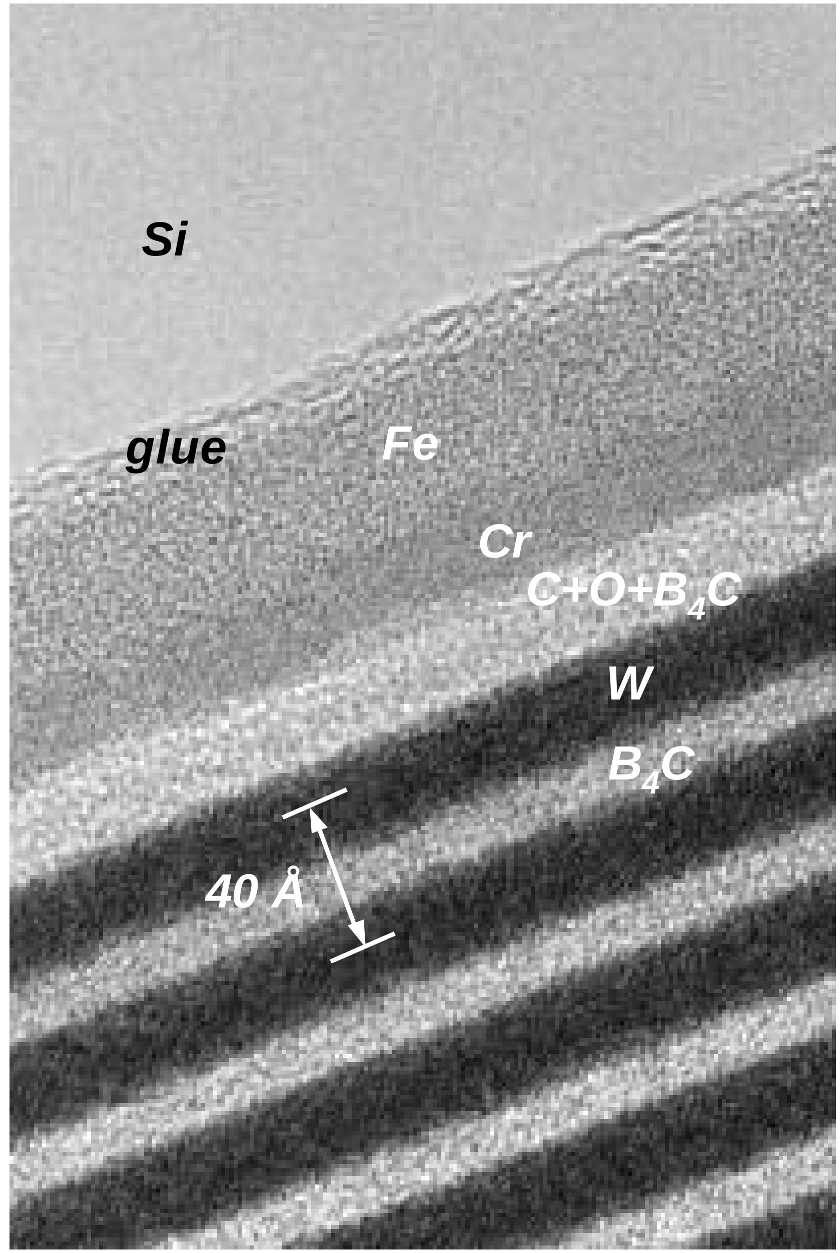
average roughness = 2.6 Å

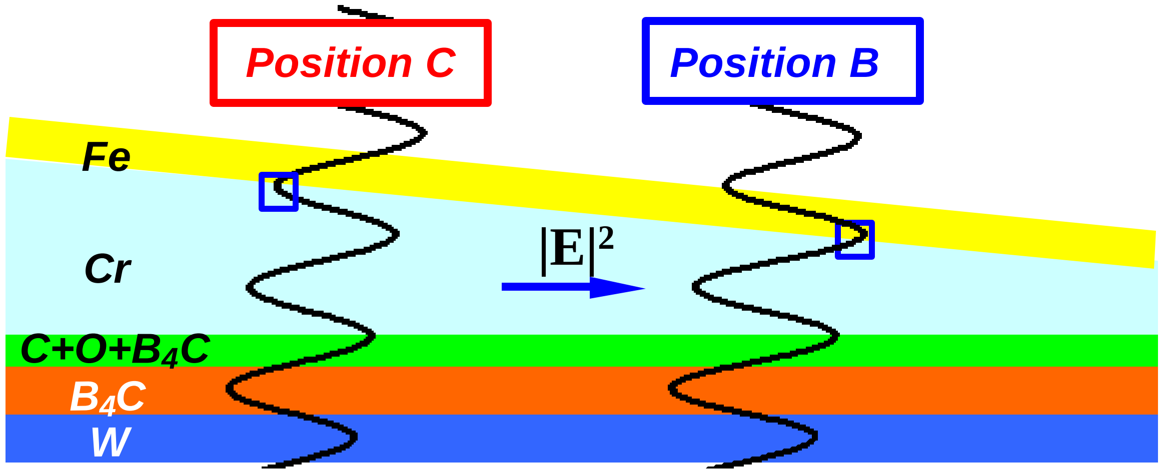
RMS roughness = 4.7 Å

Transmission Electron Microscopy Image for Fe/Cr/Multilayer SWG
(National Center for Electron Microscopy, LBNL)



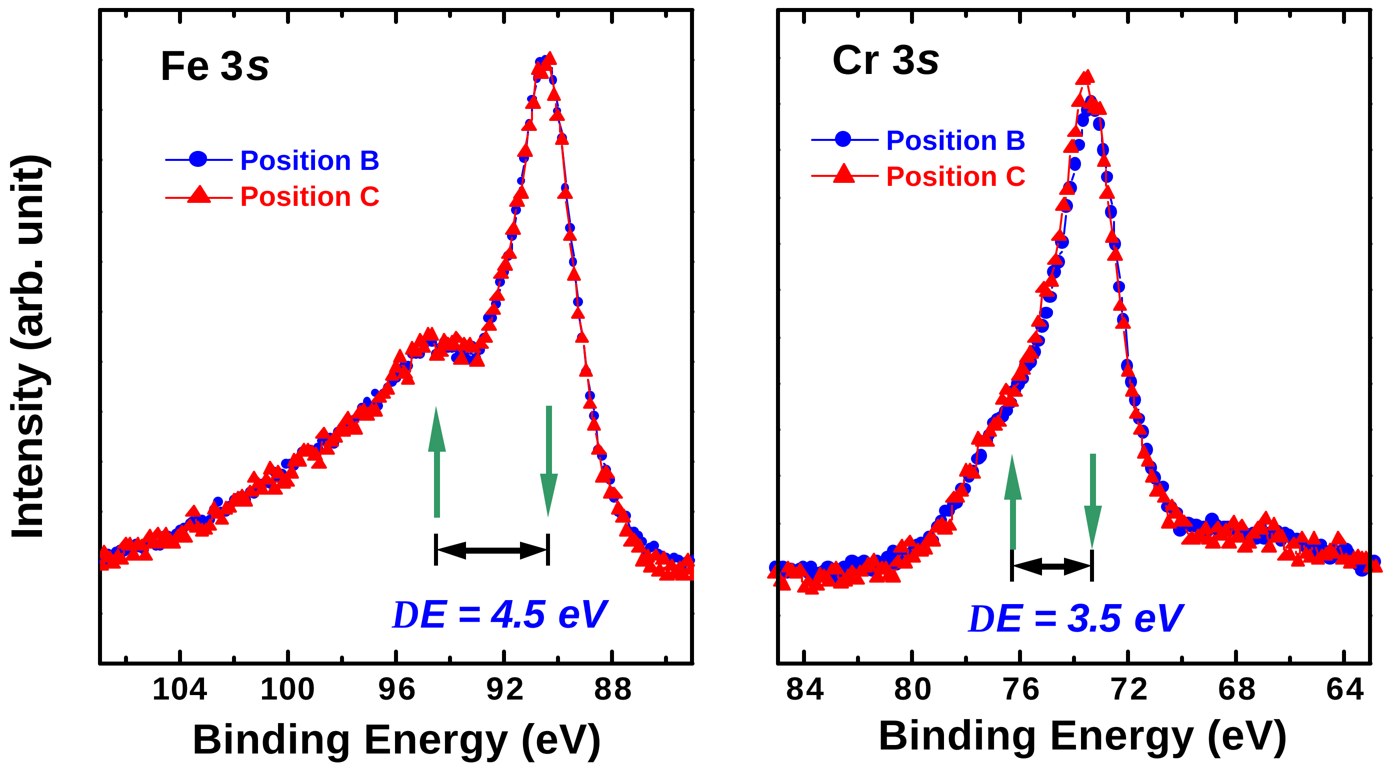






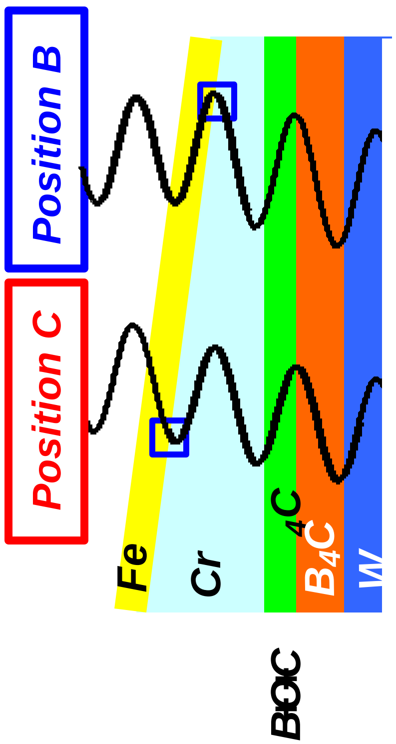
no change of local magnetic moment

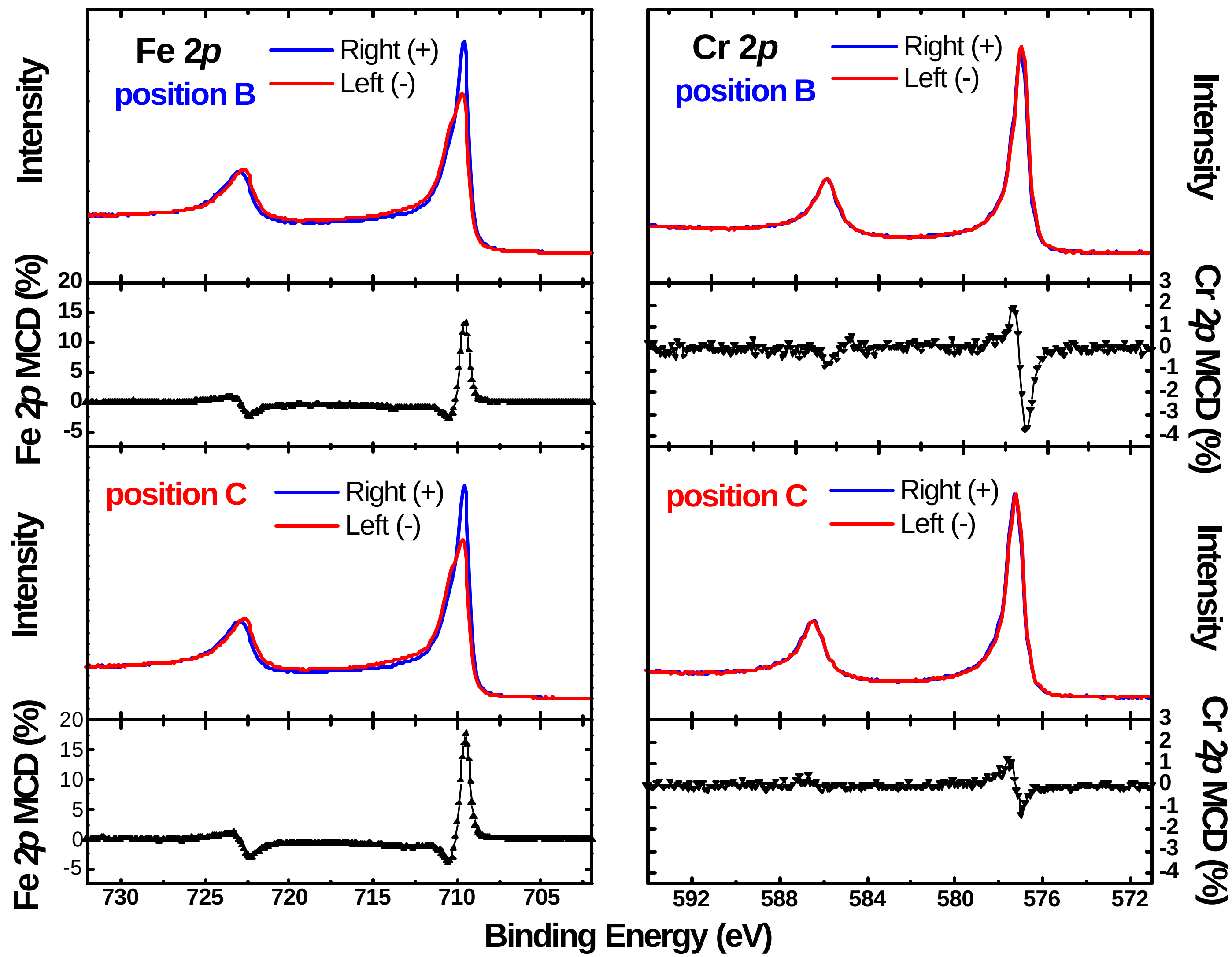
Fe and Cr 3s Photoemission in Wedge (Fe/Cr)+SWG

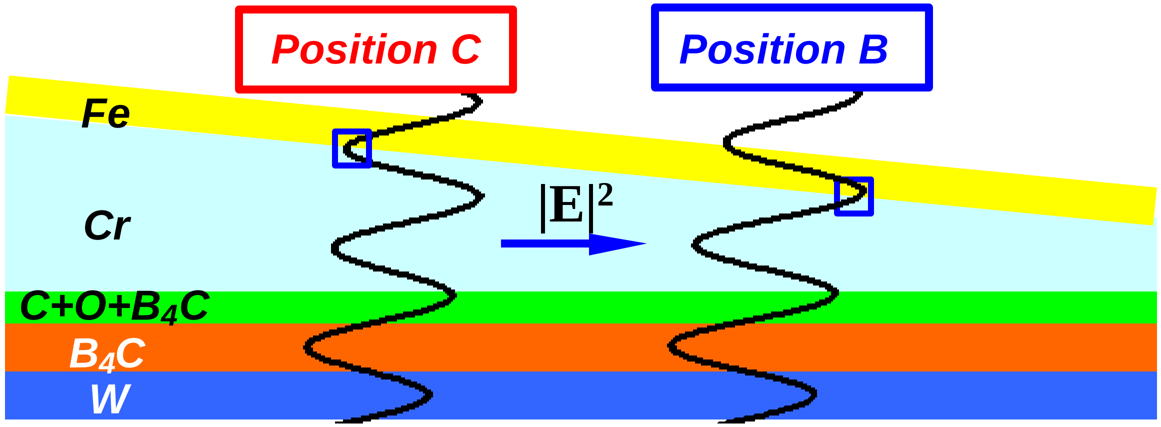


Fe & Cr 2p MCD Data for Wedge (Fe/Cr)+SWG

Cr magnetization Is antiparallel to Fe; systematic variation of MCD strengths vs d_{cr}

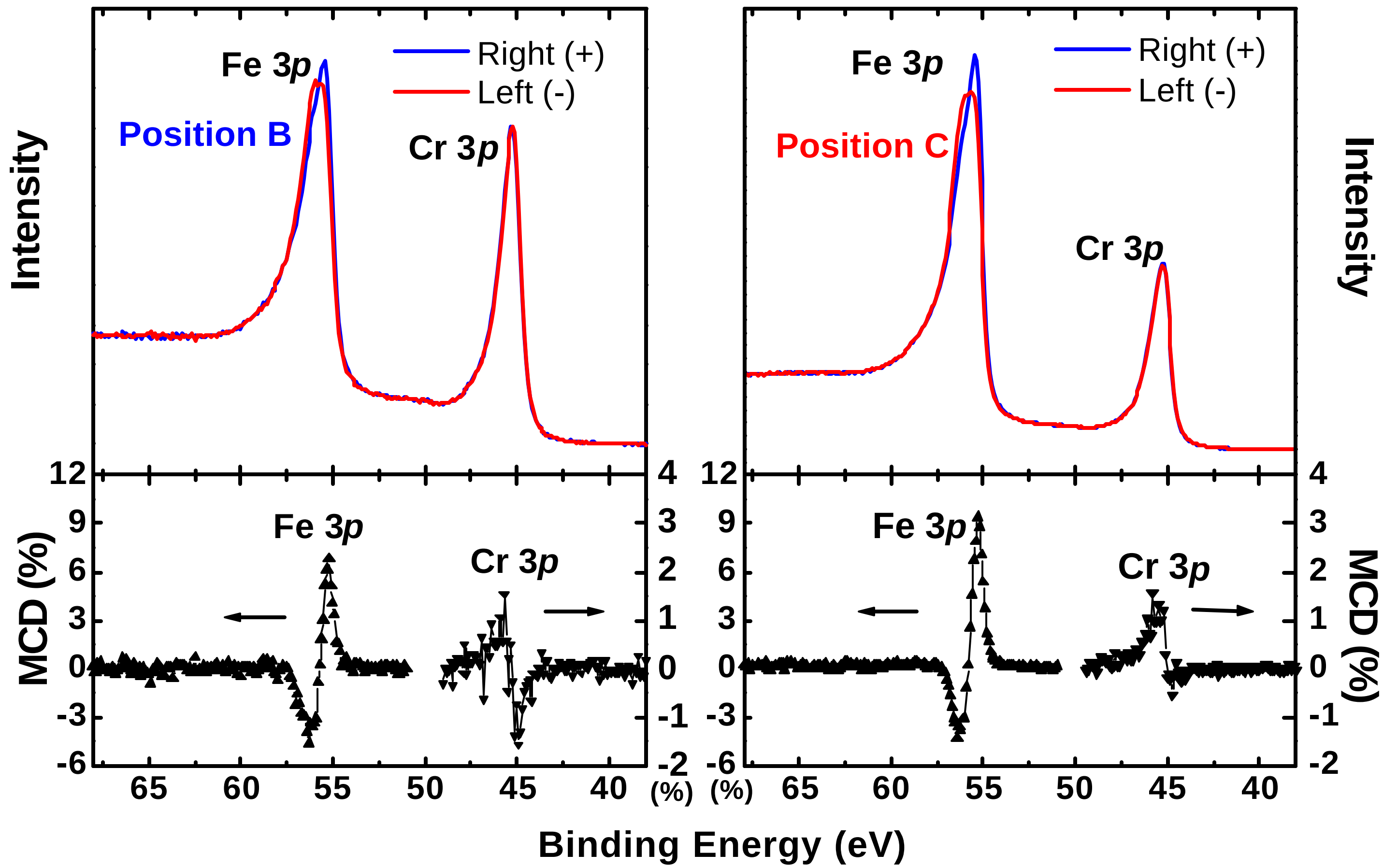


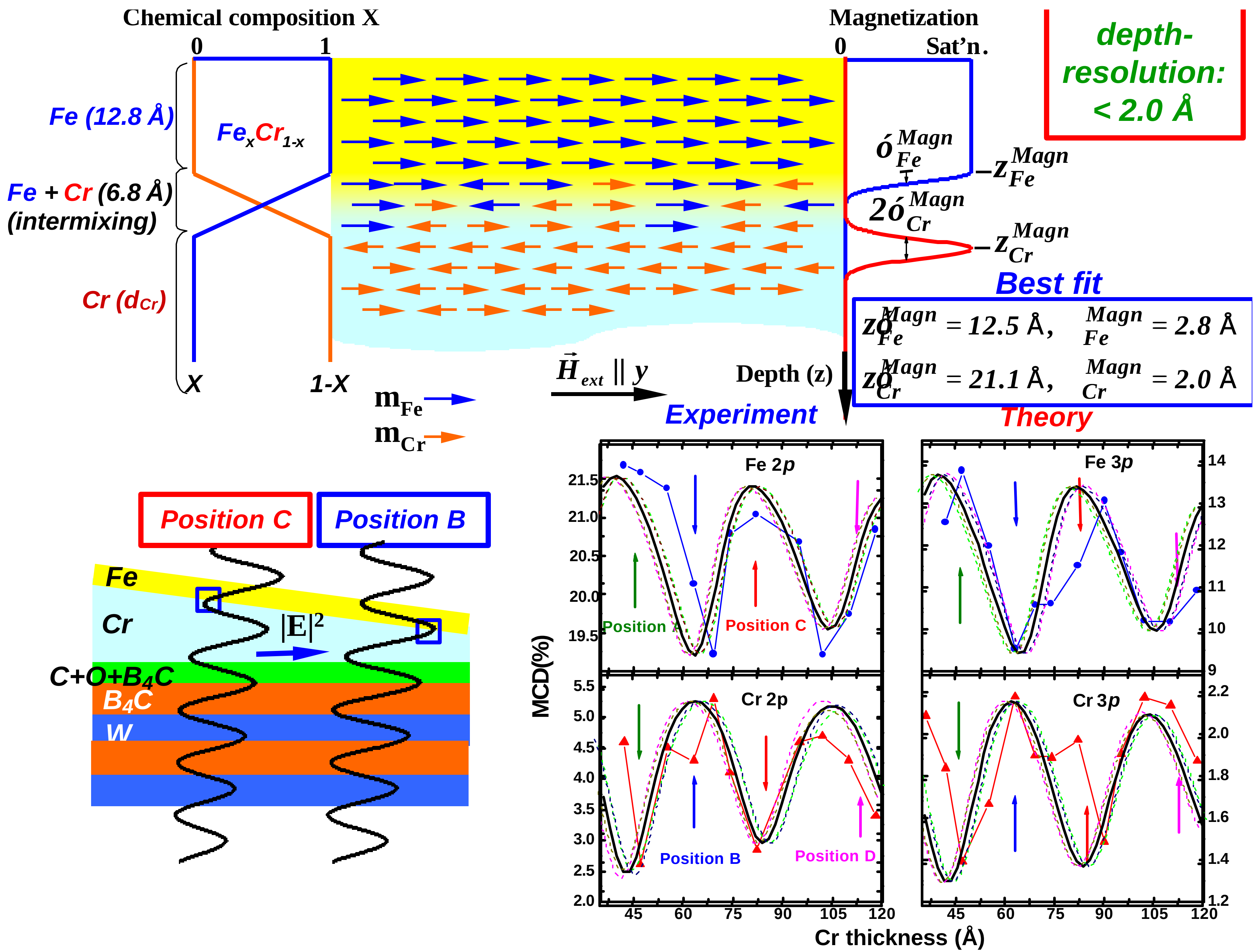




3p agrees with the results of 2p

Fe & Cr 3p MCD Expt Data for Wedge (Fe/Cr)+SWG




Chemical composition X
0
1
Fe (12.8 Å)
Fe_xCr_{1-x}
Fe + Cr (6.8 Å)
(intermixing)
Cr (d_{Cr})
X
1-X
Magnetization
0
Sat'n.
depth-resolution: < 2.0 Å
σ_{Fe}^{Magn}
z_{Fe}^{Magn}
$2\sigma_{Cr}^{Magn}$
z_{Cr}^{Magn}
Best fit
z_{Fe}^{Magn} = 12.5 Å, σ_{Fe}^{Magn} = 2.8 Å
z_{Cr}^{Magn} = 21.1 Å, σ_{Cr}^{Magn} = 2.0 Å
m_{Fe}
m_{Cr}
$\vec{H}_{ext} \parallel y$
Depth (z)
Experiment
Theory
Position C
Position B
Fe
Cr
$|E|^2$
C+O+B_4C
B_4C
W
Fe 2p
Fe 3p
Cr 2p
Cr 3p
Position A
Position C
Position B
Position D
MCD(%)
Cr thickness (Å)

Conclusion

- *depth resolved spectroscopy using multilayer standing wave generators (SWGs) promises non-destructive tool to probe buried interface in magnetic nanolayers, other nanostructures*
  - *$|E|^2$ at interface ~ about 40 % (min) « 160 % (max)*
  - *first application to Fe/Cr interface*
    - *Fe, Cr : no change in magnitude of local magnetic moment*
    - *Fe : reduced M near interface*
    - *Cr : enhanced FM order near interface antiparallel to Fe M*

- *future work*
  - *probing surface segregation or phase separation with SWG*
  - *other magnetic nanostructural superlattices on SWG (e.g. exchange bias or magnetic tunneling junction)*
  - *nanocrystals deposited on SWGs*
  - *x-ray fluorescence or x-ray scattering with overlayers on SWG*
  - *photoelectron emission microscopy (PEEM) on SWGs*

Principal Investigators, Coworkers and Collaborators

S.-H. Yang, B. S. Mun, N. Mannella, L. Zhao, A. Kay and C. S. Fadley
Materials Sciences Division, Lawrence Berkeley National Laboratory
Deparment of Physics, University of California, Davis

F. Salmassi and J. Underwood
Center for X-ray Optics, Lawrence Berkeley National Laboratory

M. Van Hove and J. Kortright
Materials Sciences Division, Lawrence Berkeley National Laboratory

S. K. Kim
Center for Nanospinics of Spintronic Materials
Korea Advanced Institute of Science and Technology, Korea

E. Arenholz, A. Young, and Z. Hussain
Advanced Light Source, Lawrence Berkeley National Laboratory

C. Y. Song, C. Kisielowski, U. Dahmen
National Center for Electron Microscopy, Lawrance Berkeley National Laboratory