Research at the Material and Chemical Sciences Beamline (9.3.2) at the ALS

Nasser M. Hamdan

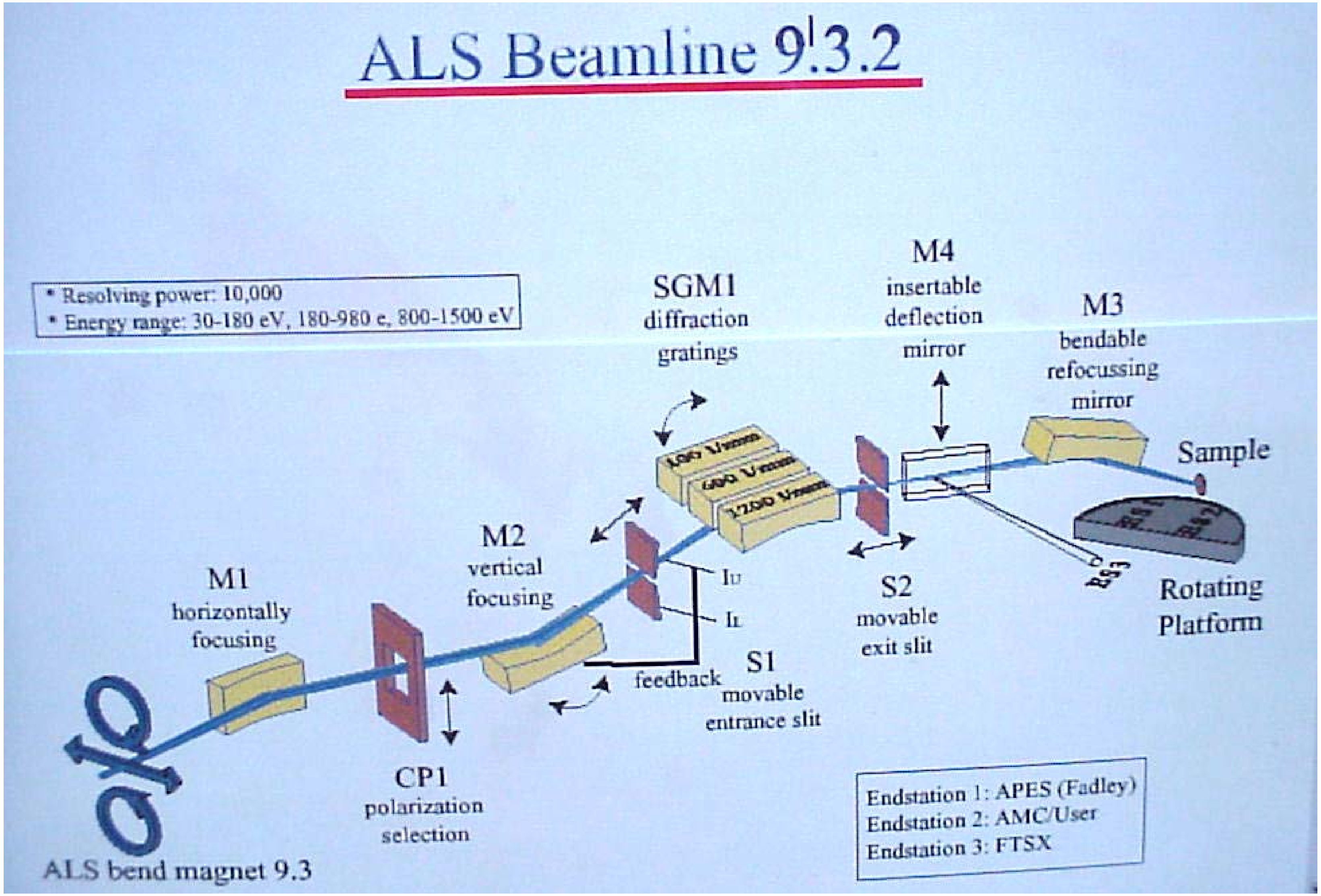
Physics Department

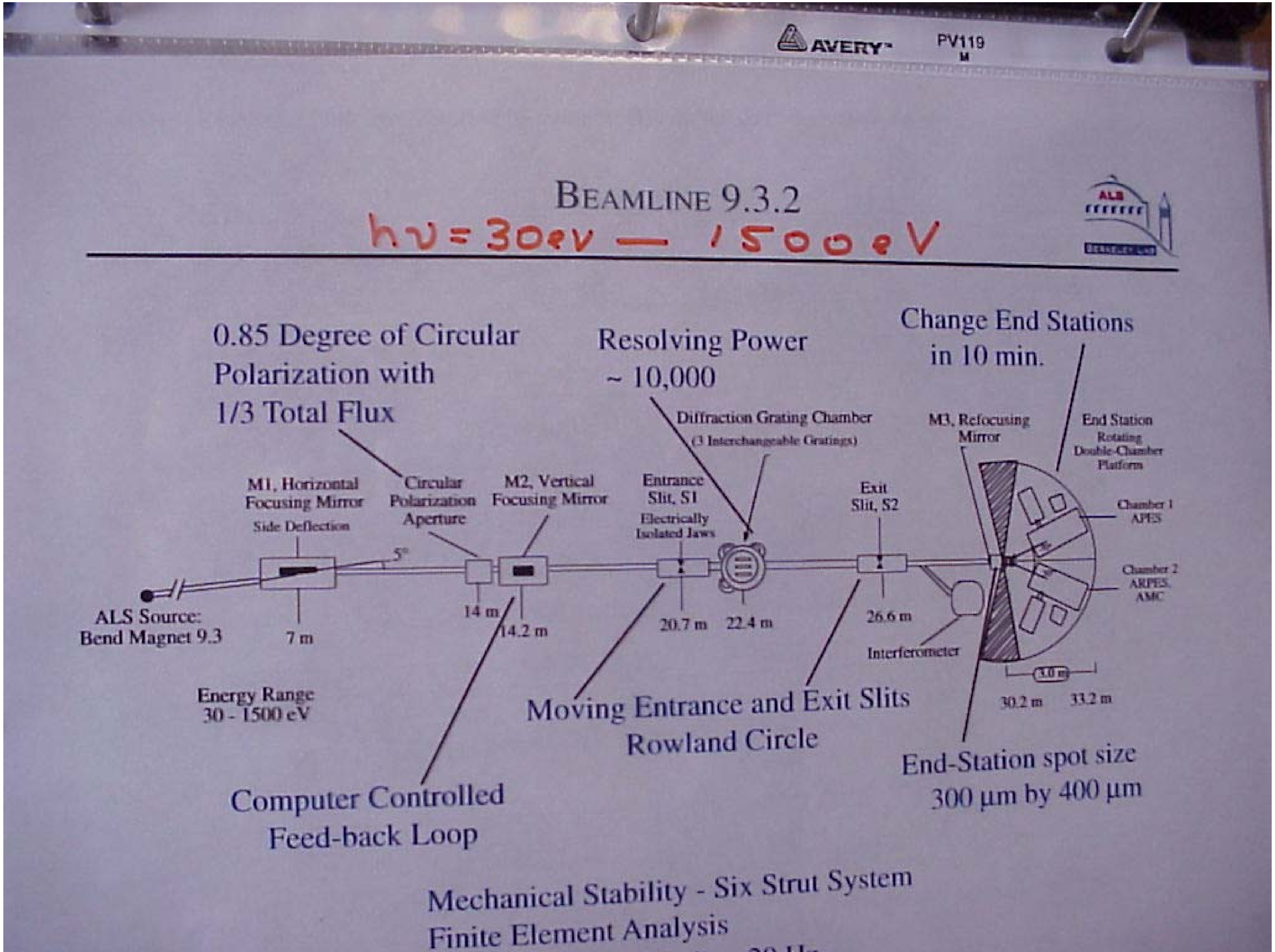
American University of Sharjah

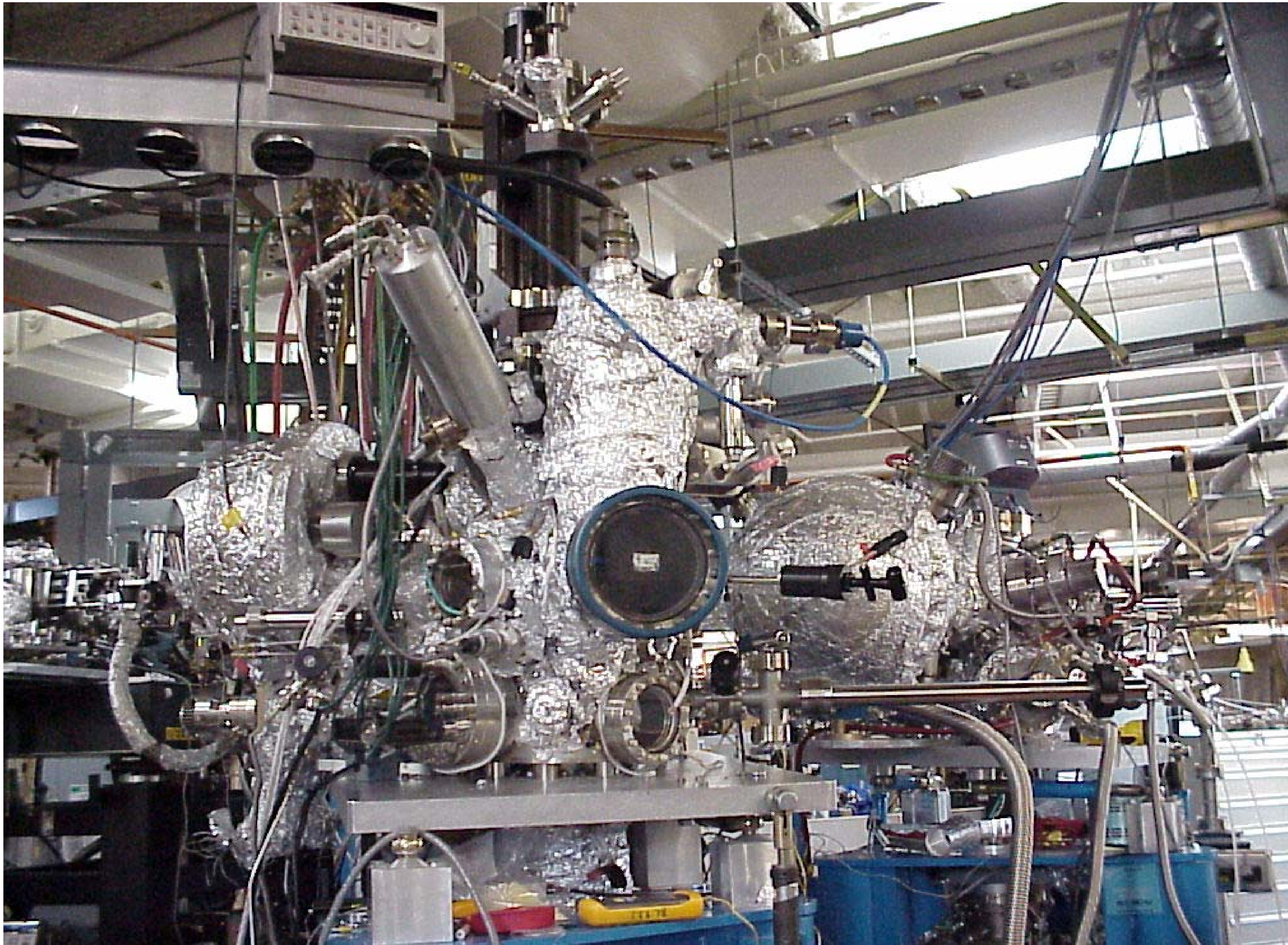
Acknowledgment: This work was co-sponsored by the DOE cooperative research program and the ALS

Talk Outline

- Introduction about the beamline and end stations.
- List of research projects performed at the beamline
- Two examples (gate oxides and high Tc superconductivity)


ALS Beamline 9.3.2
* Resolving power: 10,000
* Energy range: 30-180 eV, 180-980 e, 800-1500 eV
SGM1
diffraction
gratings
M4
insertable
deflection
mirror
M3
bendable
refocussing
mirror
Sample
M2
vertical
focusing
M1
horizontally
focusing
S2
movable
exit slit
Rotating
Platform
S1
movable
entrance slit
feedback
CP1
polarization
selection
ALS bend magnet 9.3
Endstation 1: APES (Fadley)
Endstation 2: AMC/User
Endstation 3: FTSX


AVERY
PV119
BEAMLINE 9.3.2
hν = 30eV — 1500eV
0.85 Degree of Circular Polarization with 1/3 Total Flux
Resolving Power ~ 10,000
Change End Stations in 10 min.
Diffraction Grating Chamber
(3 Interchangeable Gratings)
M3, Refocusing Mirror
End Station
Rotating Double-Chamber Platform
M1, Horizontal Focusing Mirror
Side Deflection
Circular Polarization Aperture
M2, Vertical Focusing Mirror
Entrance Slit, S1
Electrically Isolated Jaws
Exit Slit, S2
Chamber 1
APES
Chamber 2
ARPES, AMC
5°
ALS Source:
Bend Magnet 9.3
7 m
14 m
14.2 m
20.7 m
22.4 m
26.6 m
Interferometer
3.0 m
30.2 m
33.2 m
Energy Range
30 - 1500 eV
Moving Entrance and Exit Slits
Rowland Circle
End-Station spot size
300 µm by 400 µm
Computer Controlled
Feed-back Loop
Mechanical Stability - Six Strut System
Finite Element Analysis



Beamline properties

- Bend magnet, flux (1.9 GeV, 400 mA) ~10^{11} photons/s/0.1 BW at 400 eV)
- Energy range (30-1200) eV (could reach 1500 with low resolution), SGM, gratings; 100, 600, 1200 lines/mm)
- Linear and circular polarization
- High resolution ($\Delta E/E$ ~5000 typical, up to 10000)
- 3 different end station with very fast switching between end stations (less than 10 minutes)

End Stations

Advance Material Chamber (AMC)	**High pressure Spectrometer**	**Fourier Transform Spectrometer FTSX**
Atomic and electronic structure of surfaces, UHV comp. Solids up to 15 mm diameter Scienta SES 100 analyzer	Photoemission at P up to 10 Torr. PHI analyzer, 16 element multichannel detector with differentially pumped electron-transfer-lens system	R~ 1,000,000 at 60eV to resolve the double-ionizing series in helium.
Sputtering, evaporation, quartz crystal oscillator, LEED, XPS, NEXAFS, MCD, sample transfer, sample manipulator, heating, cooling,		

B.L. 9.3.2 is a Strong SESAME promoter
Visits of Scientists from the Middle East

- **Tabet** (Algeria), **Jalil** (Palestine) , **Faiz** (Sri-Lanka), (All from KFUPM, Saudi Arabia).
- **Hallak** (Betlehem University, Palestine).
- **Suzer** (Bilkent University, Turkey)
- **Abdelrahim,** (Kuwait).
- **Salim** (Pakistan, Canada).

Achieved and on going projects

- **High resolution NEXAFS investigations of High Tc superconductors:** Several HTSC systems were investigated to study the hole concentration in the CuO_2 planes.

  - γ–irradiated BSCCO 2223 : Hamdan (AUS, ALS) Faiz,(KFUPM), Hussain (ALS)
  - Fluorinated Pb-doped Hg-1223 (Hamdan and Hussain)
  - Ce substitution in Tl-1223 and Tl-1212 (Hamdan, Salim (KFUPM) and Hussain)
  - Oxygen stoichiometry in Tl-1234 (Hamdan, Faiz, Hasan (AUS), Salim and Hussain.)

Projects continued

- High resolution XPS study of oxide layers grown on Ge substrates, (Tabet, Faiz, Hamdan and Hussain).
- **"RESONANT PHOTEMISSION OF Mn AT THE L2 and L3 EDGES IN THE PREVOSKITE La0.7Sr0.3MnO3"**

  Christine Richter, Karol Hricovini and Vita Ilakovac, (University of Cergy-Pontoise, LPMS, France), N.M. Hamdan (ALS) C. Fadley, (UC Davis)

HIGH RESOLUTION XANES AND XPS INVESTIGATION ON CATALYSIS

P. Jalil, M. Faiz, N. Tabet, N.M. Hamdan, J. Diaz, Z. Hussain

- Tungestophosphoric acid ($H_3PW_{12}O_{40}$).
- Industrial catalysts:

  Micro (*Zeolite Y, ZSM-5)*

  Mesoporous Aluminosilicate catalyst, *(Al-MCM-41)*.

Projects-continued

- **Photoemission Study of Purpose-Built Nano-structured RuO2/Ru thin films on Plastic substrates for Electrocatalysis Application** (Hamdan,and *Jinghua Guo*, (ALS) *L. Vayssieres, A. Hagfeldt, S-E. Lindquist*, University of Uppsala, Sweden.)
- **Studies of self assembled monolayers and Langmuir-Blodgett films using NEXAFS and XPS** (A. Johnson, U. Nevada Las Vegas, N.M. Hamdan)
- **Assessment of nanoscale surfacial and Interfacial films on Ceramic** (Cannon, LBNL, *Yoshiya* Japan, Hamdan) two articles are under preparation.

Projects continued

- **VALENCE BAND PHOTEMISSION FROM PURE AND Sr DIFFUSED SINGLE CRYSTAL ANATASE TiO_2(001) SURFACES. S.** Thevuthasan, V. Shutthanandan, M.A. Henderson, G.S. Herman*, and S.A. Chambers, (PNL) S. Mun, **N.M. Hamdan**, D.K. Shuh, and C.S. Fadley, .
- **X-RAY MAGNETIC CIRCULAR DICROISM EXPERIMENTS IN AMORPHOUS IRON SILICIDE and COBALT SILICIDE FILMS**, j. Diaz, (Universidad de Oviedo Spain), Hamdan and Hussain (ALS)
- Study of amorphous carbon structure, Diamond and graphite using NEXAFS and EXAFS, Diaz, Hamdan and Hussain

- Soft X-ray Photoemission Studies of Hf Oxidation, Sefik Suzer (Turkey), Hussain and Hamdan.

Examples

1- NEXAFS of HTSC
(Investigation of Hole doping)

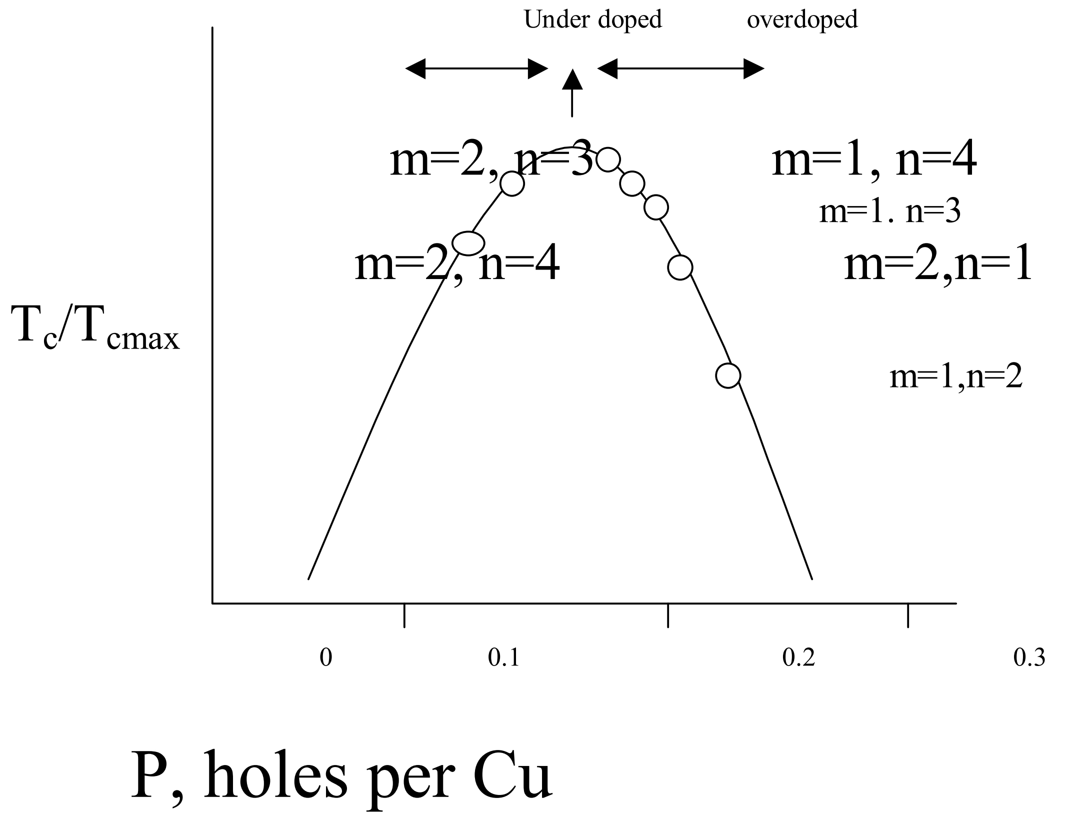
2-XPS and ARPES Study on
Gate Oxides, an Industrial
Application

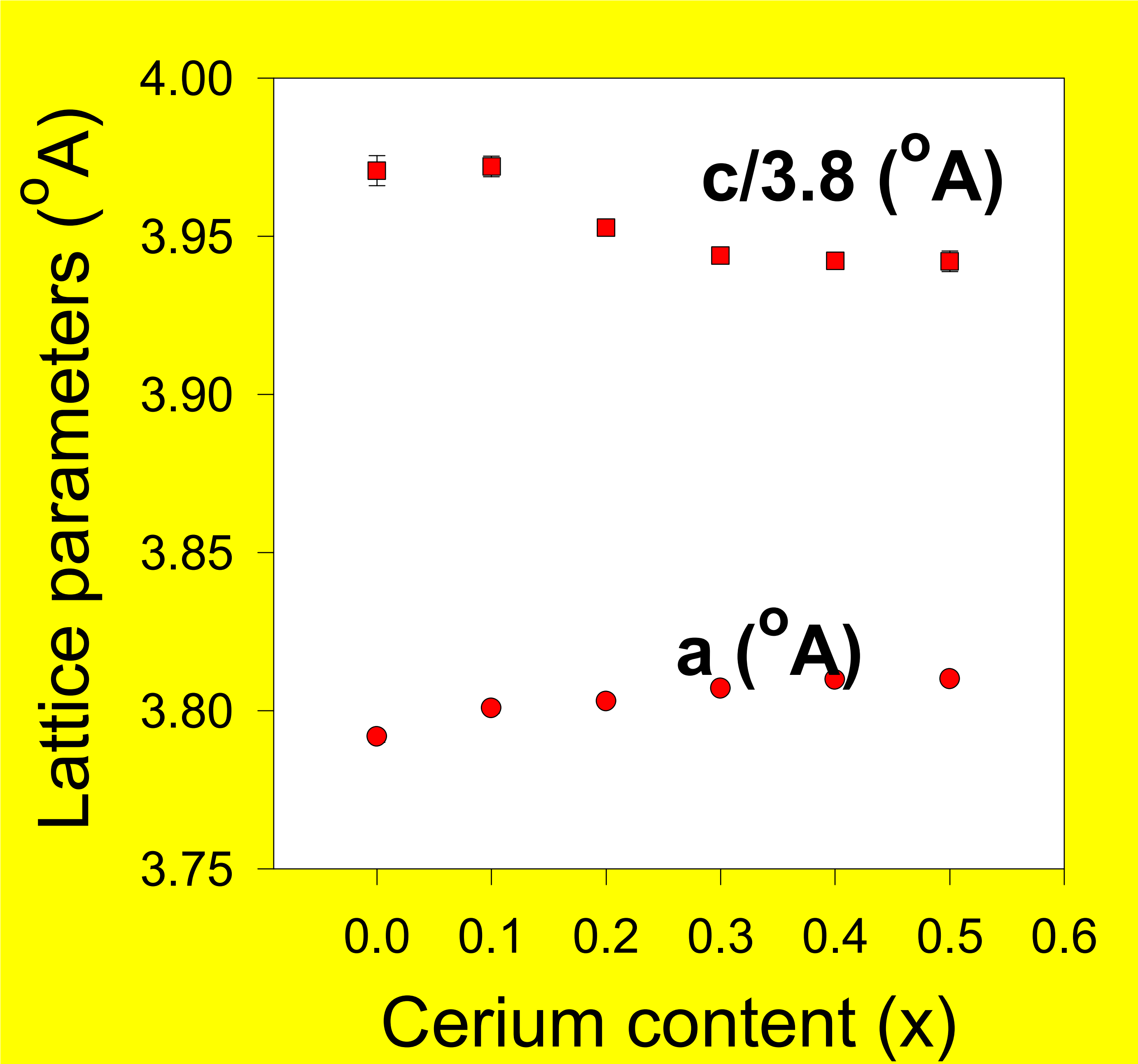
Near Edge X-ray Absorption Fine Structure Spectroscopy in HTSC

- $Tl_1Ba_2Ca_2Cu_3O_{10}$is overdoped
- To optimize the hole doping through
  - Oxygen non-stoichiometry
  - Anion Substitution **F^- for $O^=$**
  - Cation substitution
    - Y^{+3} for Ca^{+2}
    - Pb^{+4} for Tl^{+3}
    - **$Ce^{+3,4}$ for Sr^{+2}**

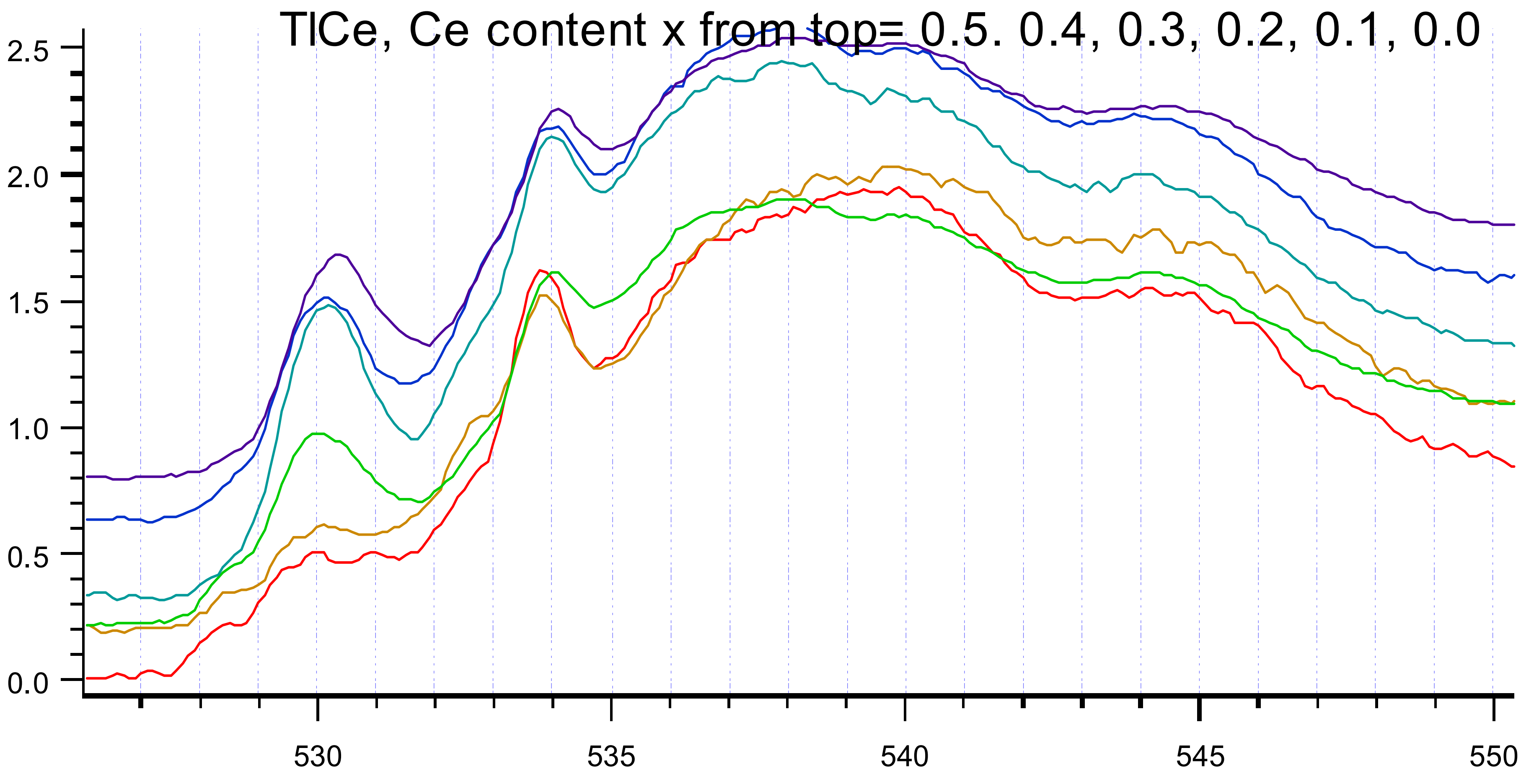
$R_mBa(Sr)_2Ca_2Cu_nO_x$

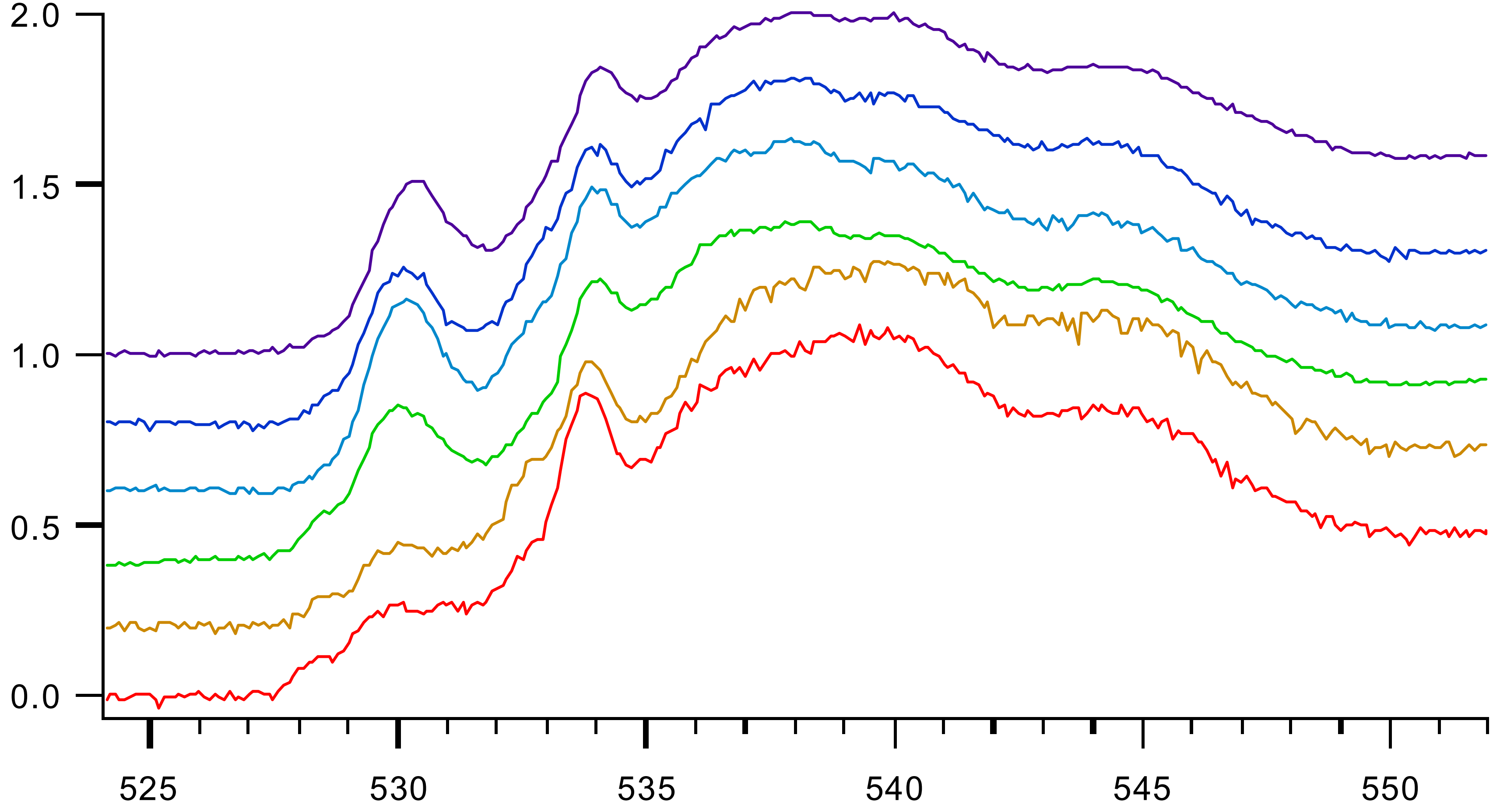
R= Hg, Tl





Lattice parameters (°A)
4.00
3.95
3.90
3.85
3.80
3.75
c/3.8 (°A)
a (°A)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
Cerium content (x)


TlCe, Ce content x from top= 0.5. 0.4, 0.3, 0.2, 0.1, 0.0
2.5
2.0
1.5
1.0
0.5
0.0
530
535
540
545
550



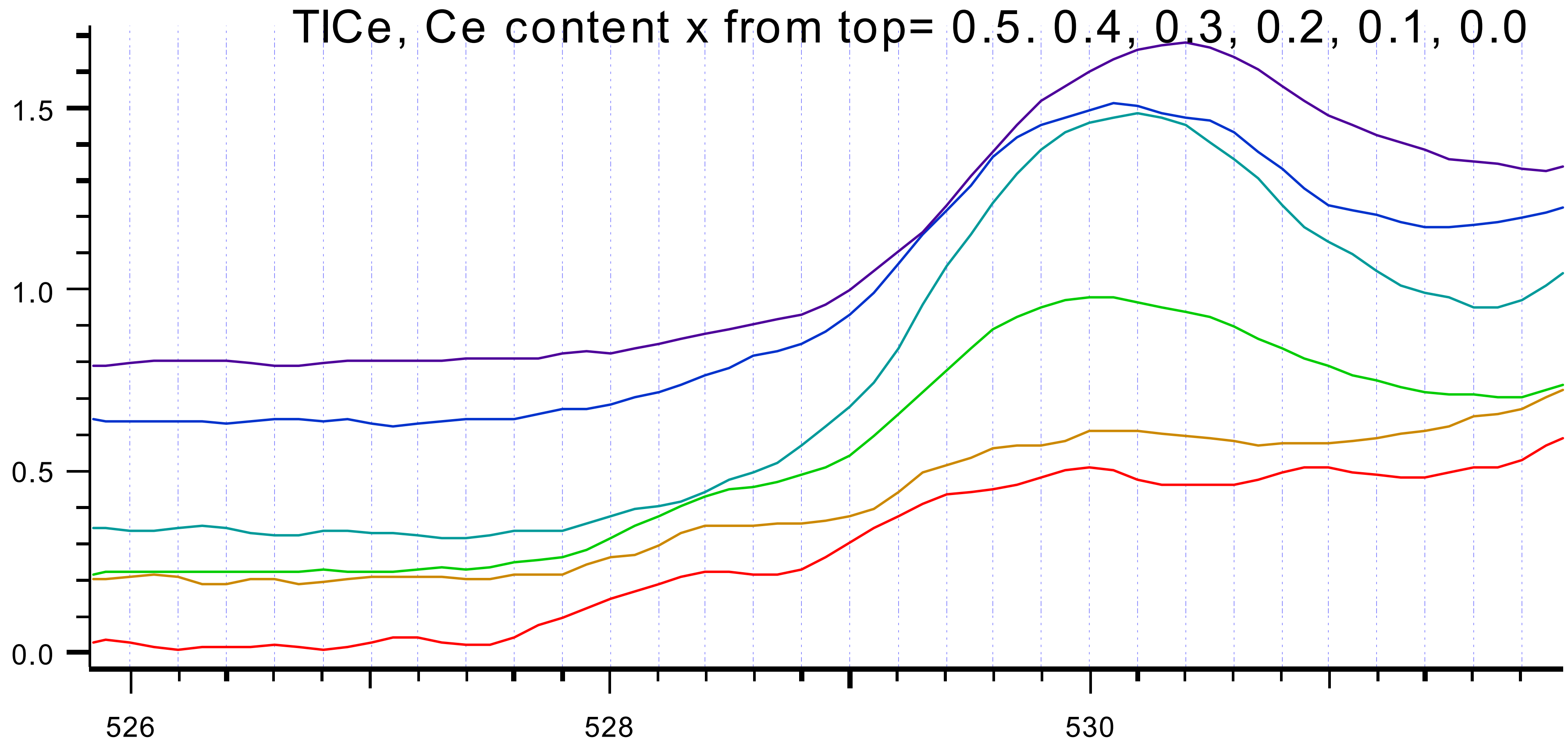
2.0
1.5
1.0
0.5
0.0
525
530
535
540
545
550

O K-edge

- Pre-edge region First peak 528.3 eV is transition from O 1s core state to to O 2p holes in the CuO_2 planes. It is directly proportional to the number of holes in CuO_2 planes.
- This assignment agrees with band structure calculations and polarized XAS measurements in single crystal HTSC.
- Intensity of this peak decreases systematically as Ce was replacing Sr, and hence decreasing the hole concentration in the CuO_2 planes near the Fermi level, and reduces the effective valence of Cu.

- The peak at ~ 530 eV is ascribed to core level excitations of O 1s electrons to empty states with mainly 2p character located in the Sr and Tl-O planes.
- That explains the drastic increase in the intensity of this peak as Ce content was increased because of the higher Ce valency.
- Another possible final state that is responsible for this peak is the upper –Hubbard band of Cu 3d states hybridized with the O 2p states.

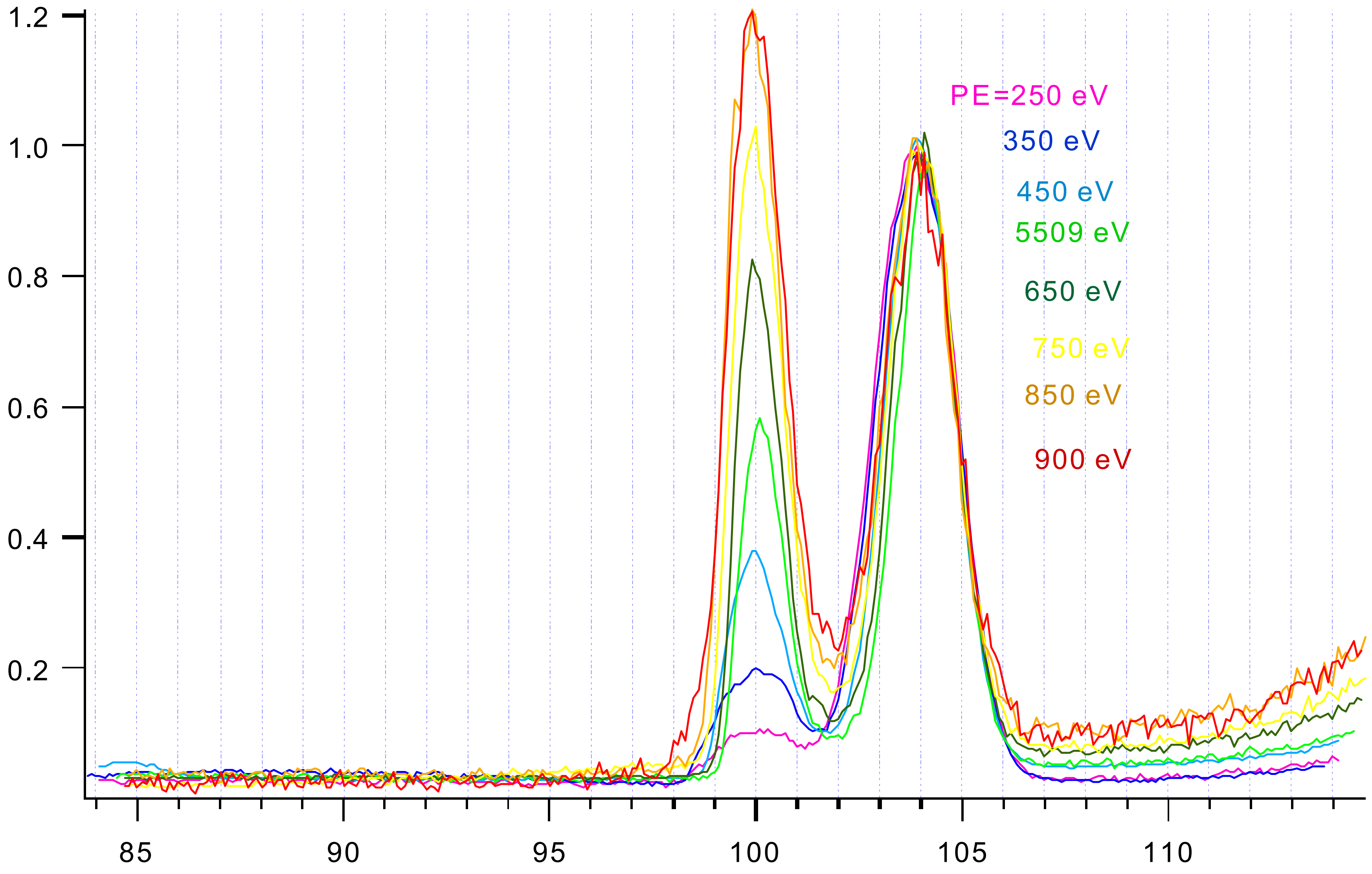
- The high energy range above the edge is mainly due to continuum absorption to Tl 6p, Sr 5d abd Ba 4f empty states hybridized with O 2p.

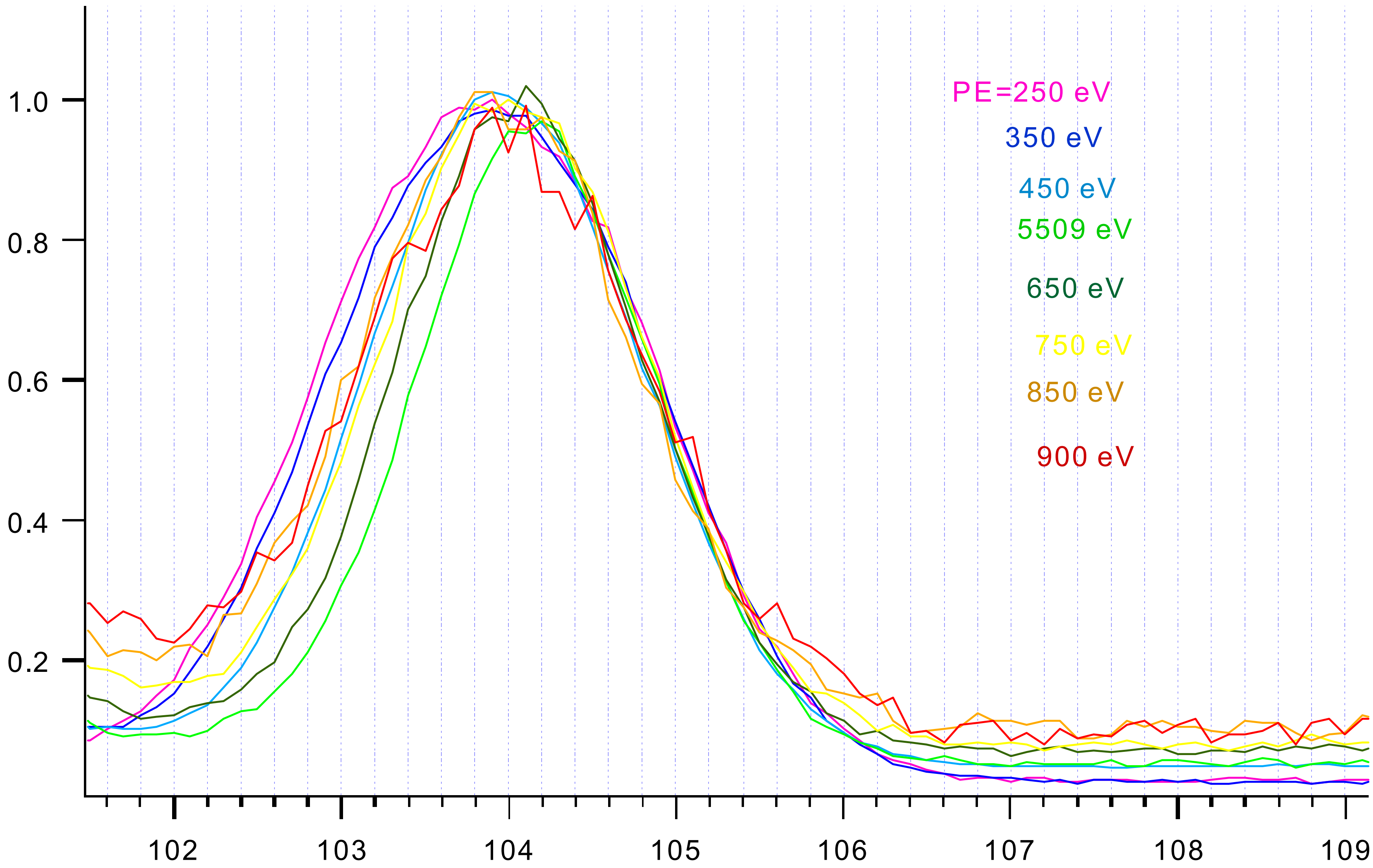

TlCe, Ce content x from top= 0.5. 0.4, 0.3, 0.2, 0.1, 0.0
1.5
1.0
0.5
0.0
526
528
530

Core level photoemission and structure in N doped Si/SiO2 gate oxides

ALS	**Applied Materials**	**UC-Davis and LBNL**
Nasser Hamdan Zahid Hussein	CR Brundle Edward Principe	Charles Fadley

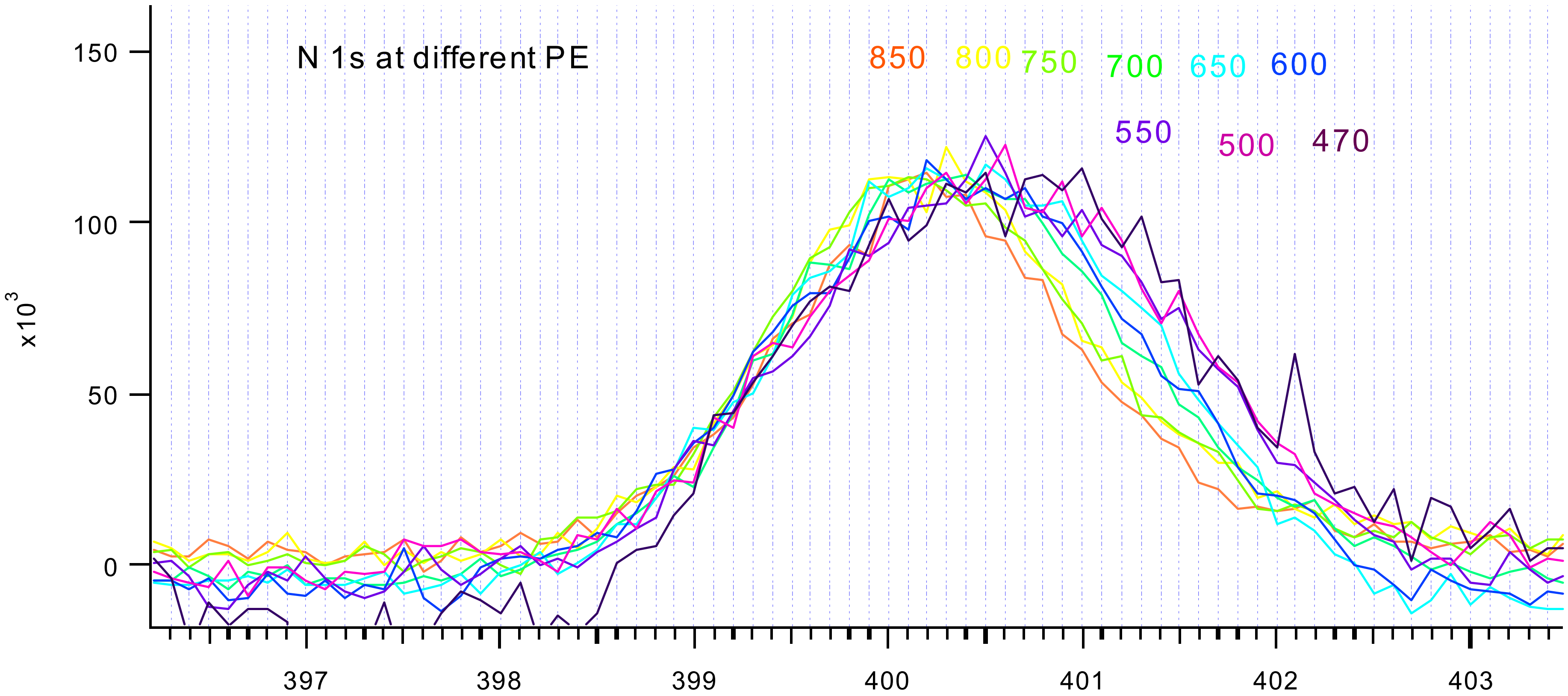
- Ultra thin films of N-SiO_2 on Si wafers (Pentium IV and V processors): AMD & Intel
- Real problems in manufacturing the wafers because of variations in the electrical properties of MOSFET gates.
- N increase K value


PE=250 eV
350 eV
450 eV
5509 eV
650 eV
750 eV
850 eV
900 eV
1.2
1.0
0.8
0.6
0.4
0.2
85
90
95
100
105
110


PE=250 eV
350 eV
450 eV
5509 eV
650 eV
750 eV
850 eV
900 eV
1.0
0.8
0.6
0.4
0.2
102
103
104
105
106
107
108
109

Si-ep

- Interested in SiO_2 peak.
- Si sub-oxide: SiO_2, SiO_xN_y, Si


N 1s at different PE
850
800
750
700
650
600
550
500
470
150
100
50
0
x10^3
397
398
399
400
401
402
403

N 1 s spectra

- Lower BE peak is due to $N \equiv Si_3$ bond
- The attribution of the weaker peak due to non-stoichiometric silicon nitride. BE of SiN_x varies by ~ 1.2 eV depending on x. (Si_3N_4). N-O bonding is excluded as the BE of NO_x is much higher.