Talk @ School on Magnetism at RRCAT, Indore 26th March, 2014

Beamline for soft x-ray absorption spectroscopy on Indus-2 and angle integrated photoelectron spectroscopy on Indus-1

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Acknowledgement

1. Dr.V.Ganesan, Centre-Director, CSR, Indore Centre
2. Dr.A.Gupta, Ex-Centre-Director, CSR Indore Centre
3. Dr.A.Banerjee, Scientist, CSR, Indore
4. Dr.R.J.Choudhary, Scientist, CSR, Indore
5. Dr. Mukul Gupta, Scientist, CSR, Indore
6. Dr.Dinesh Shukla, Scientist, CSR, Indore
7. Users of AIPES beamline on Indus-1
8. Dr. G.S.Lodha, and his team ISUD, RRCAT

Plan of the talk

Polarised light beamline on Indus-2

i) Design and development

ii) Experimental station for MCD study

iii) Proposed study

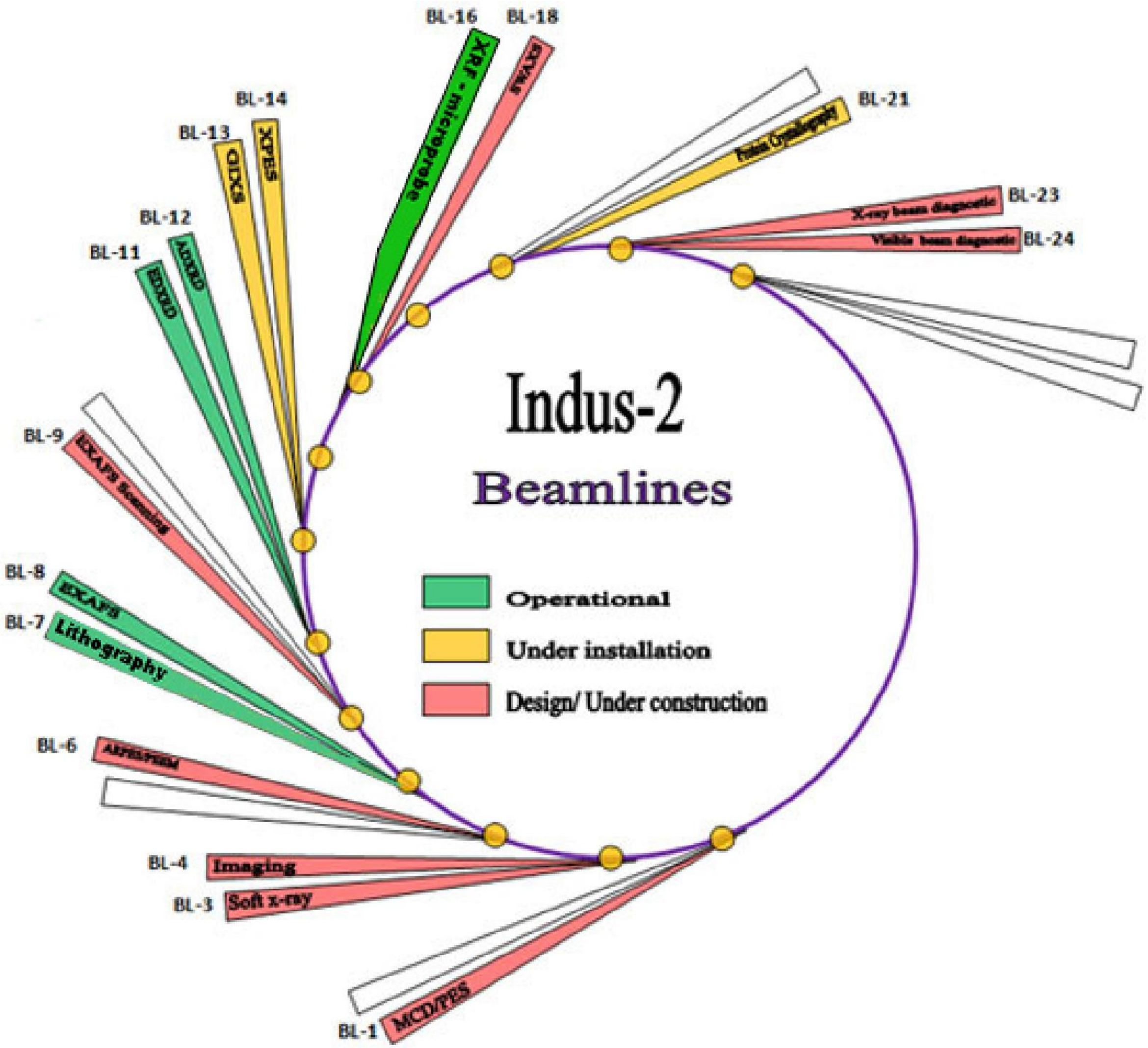
AIPES beamline on Indus-1 :

i) Beamline details

ii) Usage : PES, VBS and Band offset measurements

iii) Resonant Photoemission spectroscopy : A case study of dilute magnetic semiconductor - Fe doped TiO_2

Polarized light beamline for soft X-ray absorption study on Indus-2 developed by UGC-DAE CSR



Indus-2 storage ring
2.5 GeV, 300 mA

Polarised light beamline on Indus-2
for
Soft x-ray absorption spectroscopy

Objective :

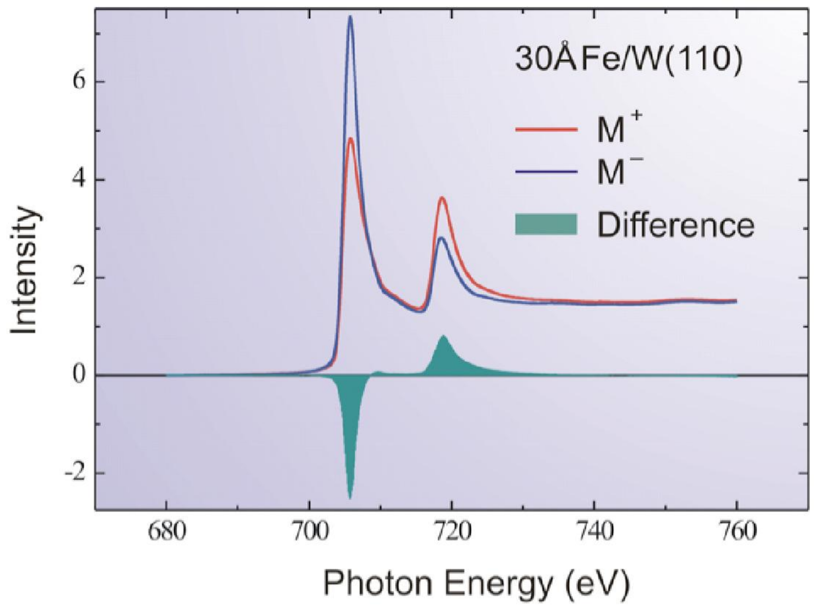
The development of SXAbsorption spectroscopy beamline on bending magnet of Indus–2 synchrotron storage ring for X-ray Magnetic Circular Dichroism (XMCD) experiment.

The developed facility will be used to investigate the microscopic origin of magnetic properties of materials through photoabsorption experiment, which are not possible, by any conventional technique.

Thus this development will provide a unique and powerful facility in India to the entire community of scientists engaged in the research on magnetism and magnetic material and thereby promote development of magnetic materials in the country.

XMCD

X-ray magnetic circular dichroism (XMCD) is a difference spectrum of two x-ray absorption spectra (XAS) taken in a magnetic field, one taken with left circularly polarized light, and one with right circularly polarized light. By closely analyzing the difference in the XMCD spectrum, information can be obtained on the magnetic properties of the atom, such as its spin and orbital magnetic moment



XMCD advantages

.This method is **very sensitive** even for the systems having small amount of magnetic atoms like magnetic sub monolayer films, nanostructures and dilute magnetic systems.

.To determine separately the contribution to magnetic moment by different constituting elements of magnetic system, i.e. **element specificity**.

. Further, **retrieve the orbital and spin contribution** to the magnetic moment of different elements present in the material.

. Microscopic origin of **magnetic anisotropy** can be determined.

Basic requirement for MCD

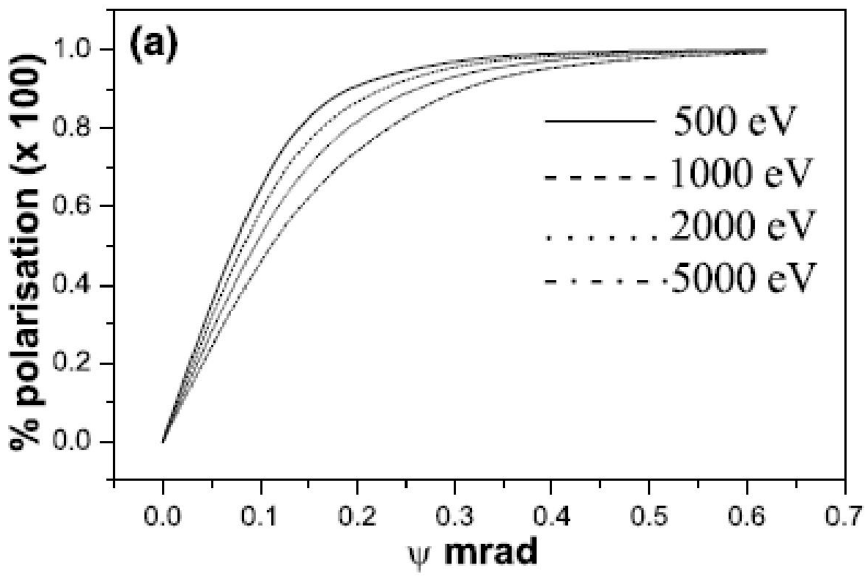
Circularly polarised x-ray source :

a) **Either from bending magnet -**
broader energy range, less photon flux,
moderate % polarisation, simple operation

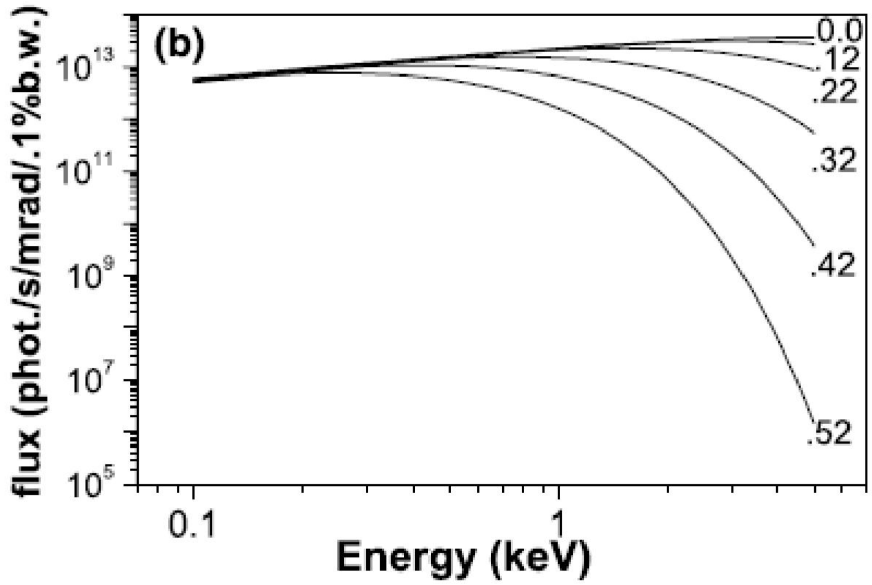
b) **Insertion devices like wiggler or undulator-**
intense photon flux, narrow photon energy in single setting,
highest % polarisation, complicate in operation

One can obtain good quality MCD spectra using bending magnet beamline

Optimisation of % polarisation and photon flux available at bending magnet port:



% polarisation Vs. viewing angle at different photon energy

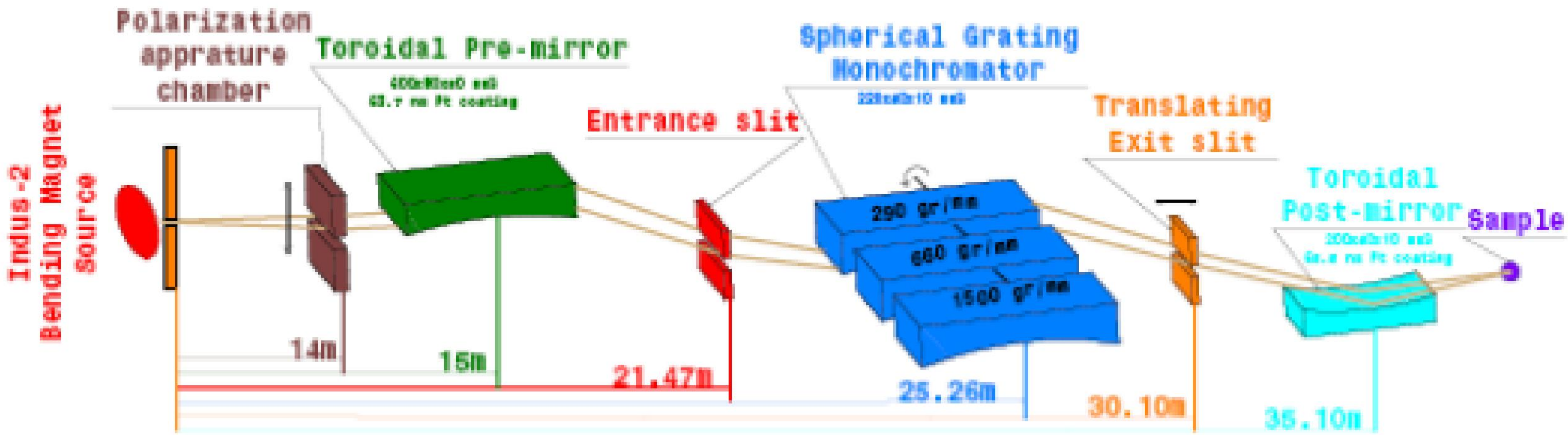


Photon flux Vs. photon energy at different viewing angle

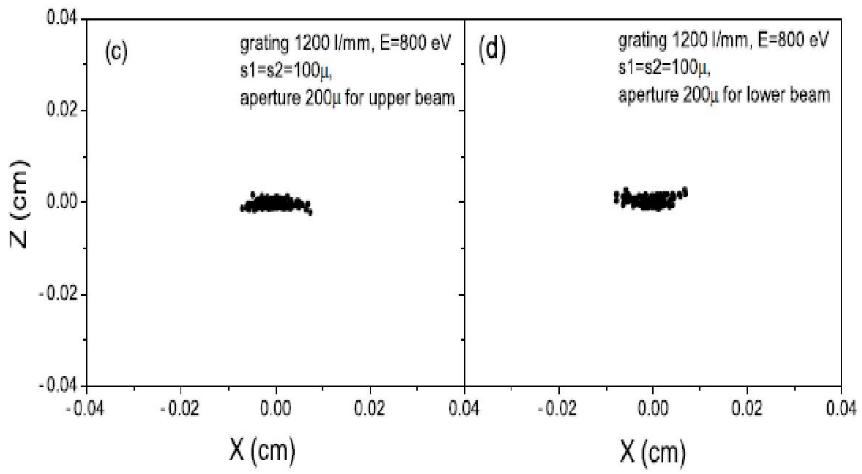
Beamline design

Beamline is designed by team of scientist of UGC-DAE Consortium for scientific research, Indore

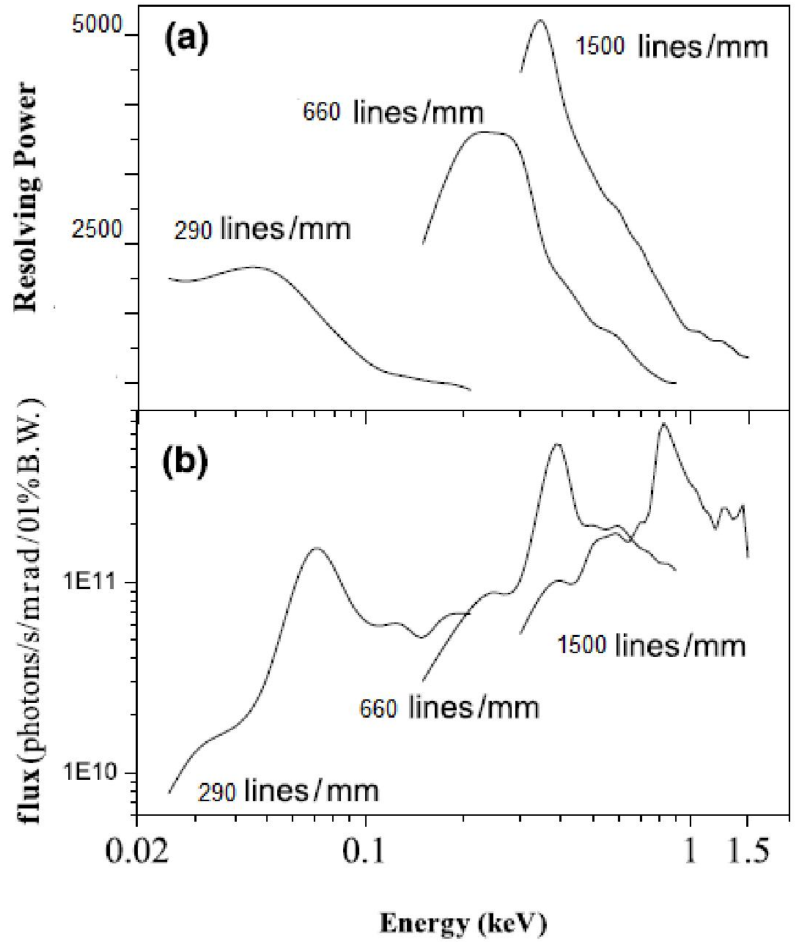
[Ref.:- Nucl.Instru.Meth. B 199 (2003) 520]



Ray tracing results:



Final simulated spot for left circular
And Right circular beam

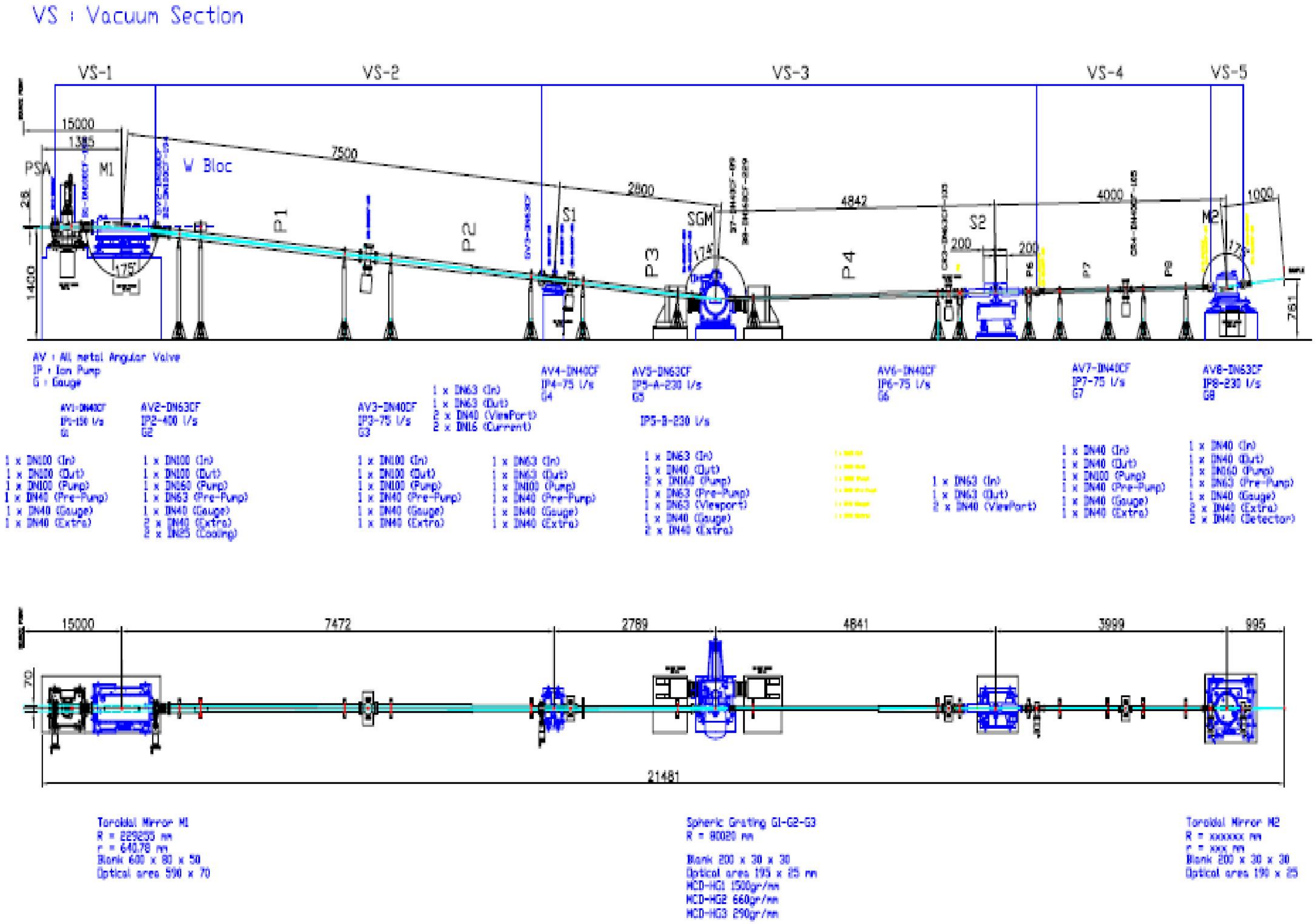


Calculated variation of resolution and photon flux as a function photon energy

For details see

Nuclear Instruments and Meth. in Physics B199 (2003) 520–525

Engineering Drawing



Specifications

Source : Bending magnet of Indus-II

Acceptance: 5 mrad horizontal $\times$1.5 mrad vertical.

Polarisation selection : Fixed Polarization selection aperture chamber including stand with four working positions:

Pre Mirror: Water cooled toroidal mirror to focus the SR beam vertically on the entrance slit and horizontally on exit slit,

Slit : Fixed entrance slit and movable exit slit

Monochromator: Spherical grating monochromator

Post Mirror : Toroidal mirror to refocus the monochromatic beam from the exit slit to a sample

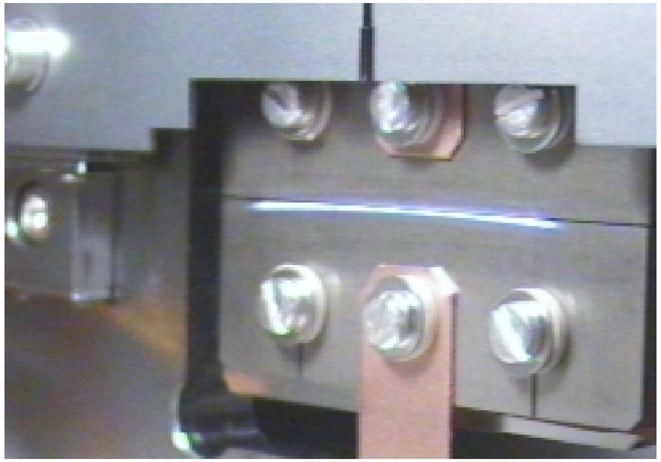
Energy Range: 100 eV to 1200 eV

Experiments: XAS, XMCD and XMLD

Commissioning Results:

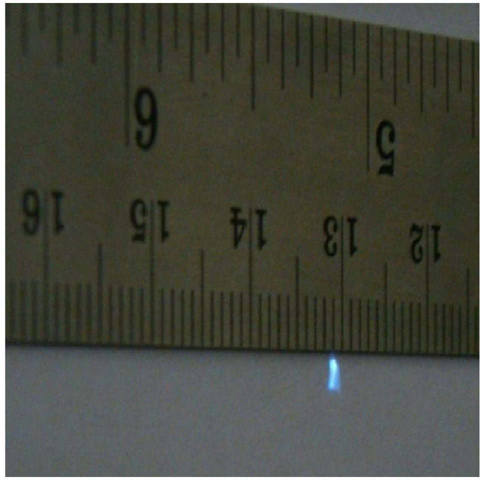
Observed beam spot

after pre-mirror/at entrance slit

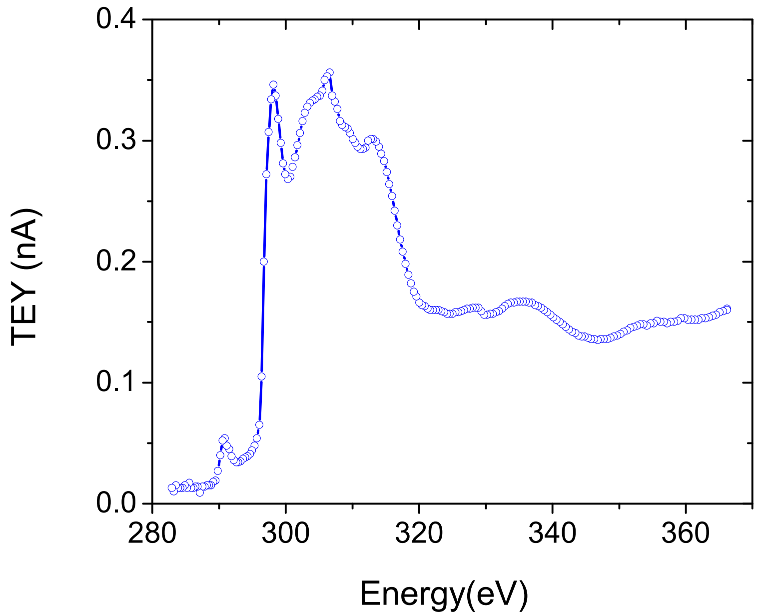


after grating/at exit slit

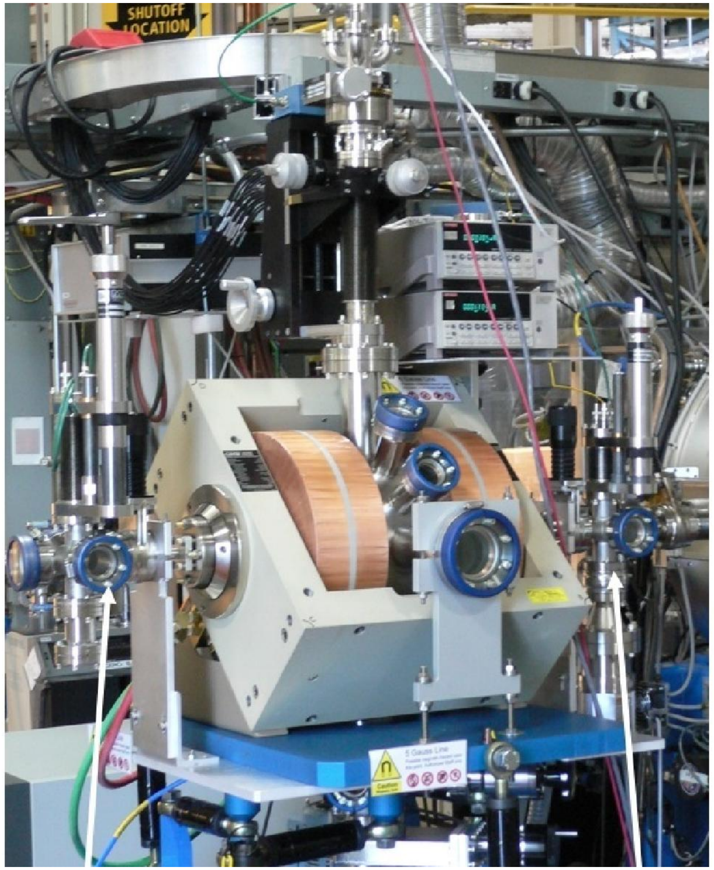
Focused spot at sample

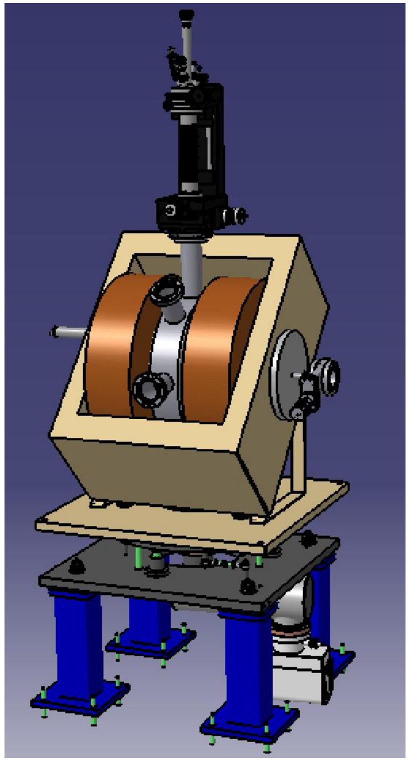


First X-ray Absorption spectrum of graphite recorded on May 17th, 2013

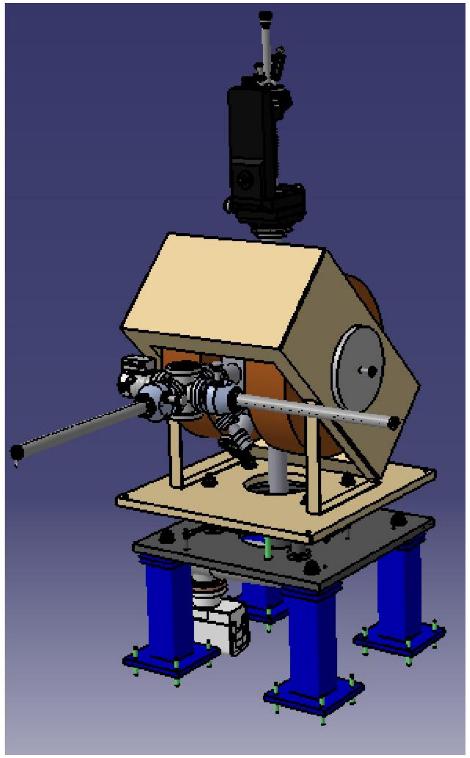


Experimental station for XMCD/XMLD type of experiment
Under development





Front



Rear

Features:

- Electromagnet : maximum field ~ 2.1 T
  (field reversal time ~ 0.2 Sec)

- Temperature: CCR based cooling (4.2 K to 400 K)

- Motorized sample stages; X Y Z translation, sample rotation around manipulator axis, possibility of azimuth rotation.

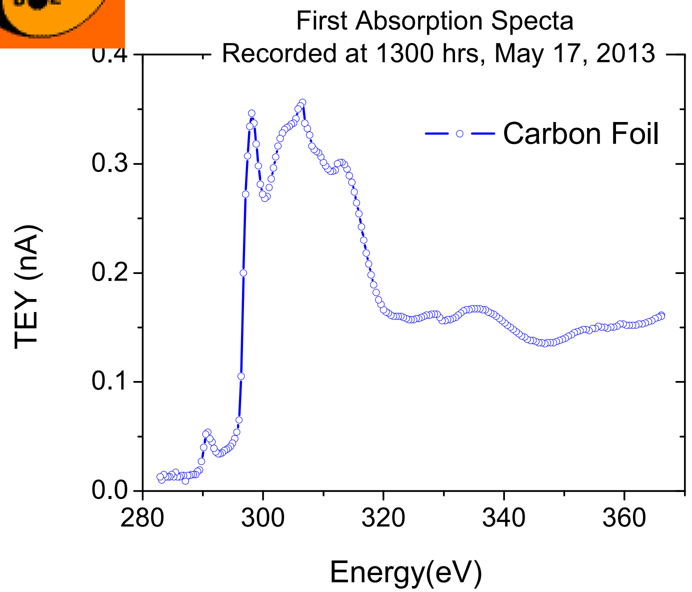
- Measurement modes: TEY and TFY mode.

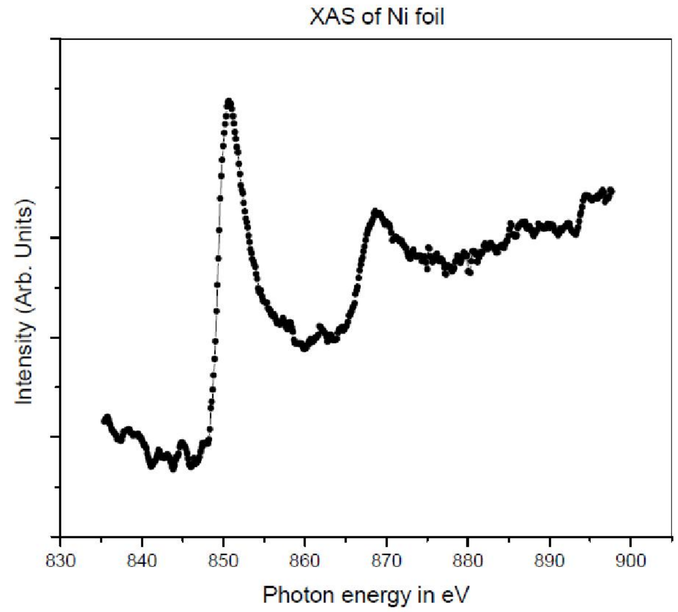
Proposed study

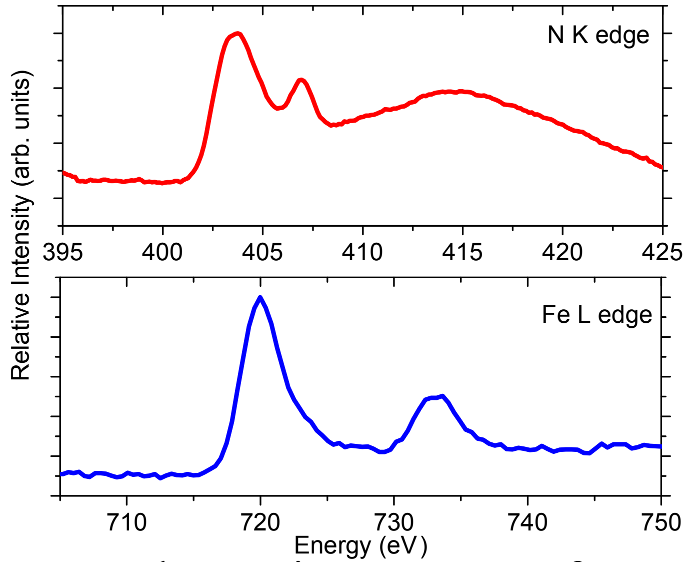
In a very broad perspective, we proposed to study:

- Complex material systems such as: doped and undoped mangnite materials, CMR materials, charge order compounds etc.
- Amorphous alloys like Fe-Zr, Ti-Ni, and Fe-Ni-Cr etc.
- Transition metals multilayer structures like Fe/Tb, Fe/Gd, and Co/Rh, Fe/Ni, Ti/Ni etc.
- Nanostructure of rare earth and transition elements such as quantum dots, nano wires etc.
- To find out magnetic dimensionality cross over from 1D-2D-3D by in- situ atom-by-atom growth.

Some XAS results



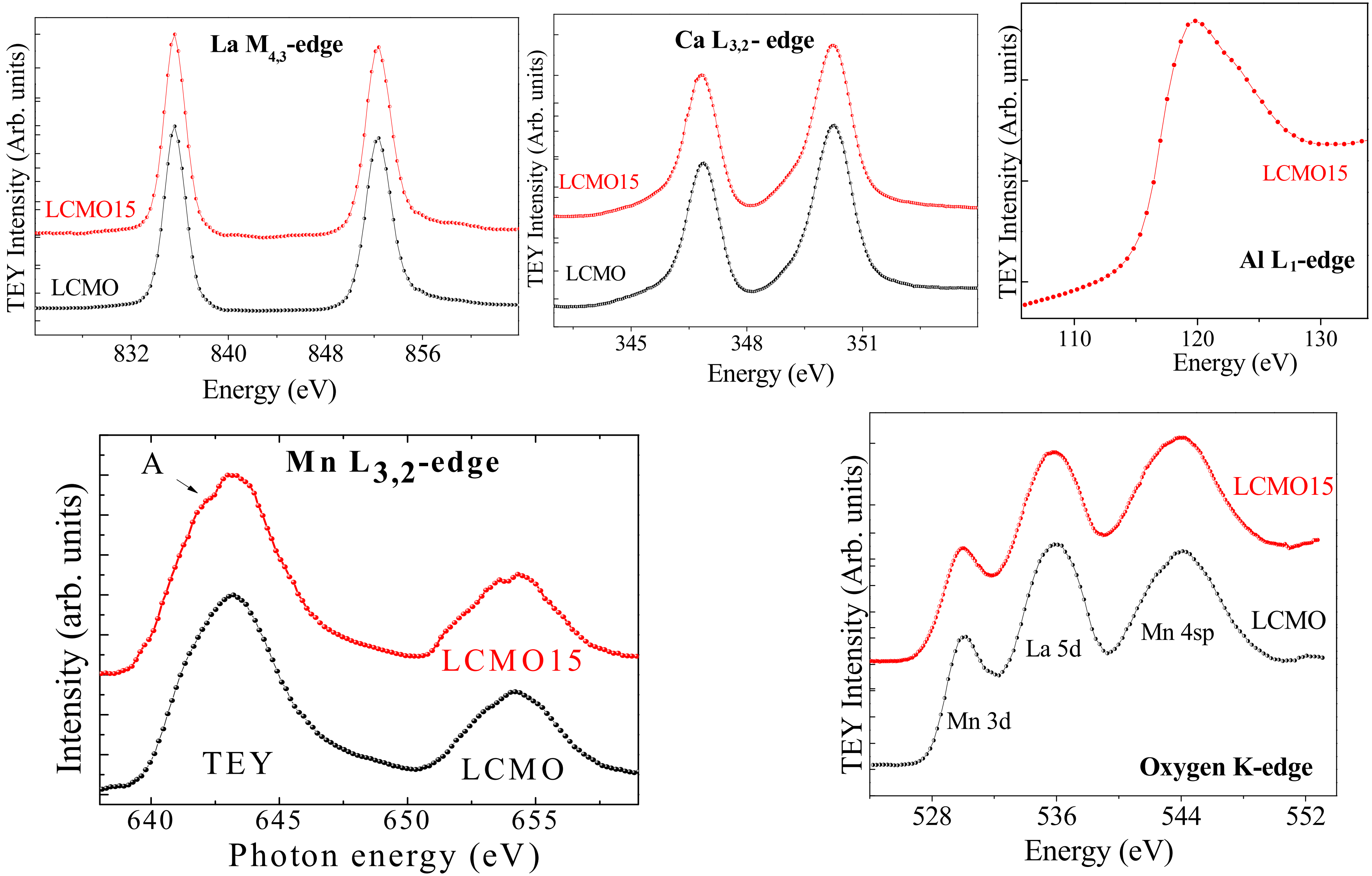




Soft X-ray absorption spectra of FeN thin films

Some XAS results

Al doped LCMO manganite thin film



Part –II : AIPES Beam line on Indus-1

. Beamline designed and developed at UGC-DAE Consortium for scientific research, Indore.
. Beamline successfully installed on Indus-1 in the year November, 2000

Beamline specifications

Source-	Bending Magnet of Indus-1
Energy	10 eV- 200 eV
Acceptance:	10 mrad horizontal x2.5 mrad vertical.
Monochromator	TGM-2600 toroidal grating monochromator, total deflection $2\theta=162$
Gratings	three interchangeable under vacuum.
Spot size	typically 5 mm ϕ
Spectral Resolution	500

Experimental station

New commercial spectrometer installed in 2004

Specifications of PES Workstation

Experimental station consists of :

- EA 125 180^0 hemispherical analyzer
- Sample preparation chamber
- Electronic detection and data acquisition system
- Argon ion gun for depth profiling study of samples.
- A diamond file to scrap the pallated samples.
- Twin anode x-ray source (Mg Kα, Al Kα) for XPS measurements.
- Sample heating (350^0C) and cooling (upto LN_2) facility.
- Magnetic sample transfer mechanism to transfer the sample from one chamber to experimental chamber without breaking the vacuum.

Measured Resolutions of the spectrometer

1. With XPS source 0.8eV at 30eV pass energy

2. SR source: Monochromator slit S1 = S2 = 300μ

Pass energy	ΔE
30eV	0.65 eV
20eV	0.45 eV
10eV	0.3 eV

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Ph.D. thesis

1.	Rahul Purandare	Pune University (2006)
2.	Shilpa Tripathi	DAVV, Indore (2008)
3.	Anupam Sharma	DAVV, Indore (2008)
4.	Ranjeet Brajpuriya	DAVV, Indore (2008)
5.	Rashi Nathawat,	Rajasthan University (2009)
6.	Ram Prakash	DAVV, Indore (2010)
7.	Amit Khare	Barkatullah Univ., Bhopal (2011)
8.	Alka Deshmukh	Pune University (2012)
9.	Shashi Shinde,	Pune University (2013)
10.	Komal Bapna	DAVV, Indore (2013)
11.	Sandeep Dhobale	Fergusson College (2013)

IOP PUBLISHING JOURNAL OF PHYSICS: CONDENSED MATTER
J. Phys.: Condens. Matter 20 (2008) 335225 (4pp) doi:10.1088/0953-8984/20/33/335225

Electronic structure of Fe (0–5 at.%) doped MoO_2 thin films studied by resonant photoemission spectroscopy

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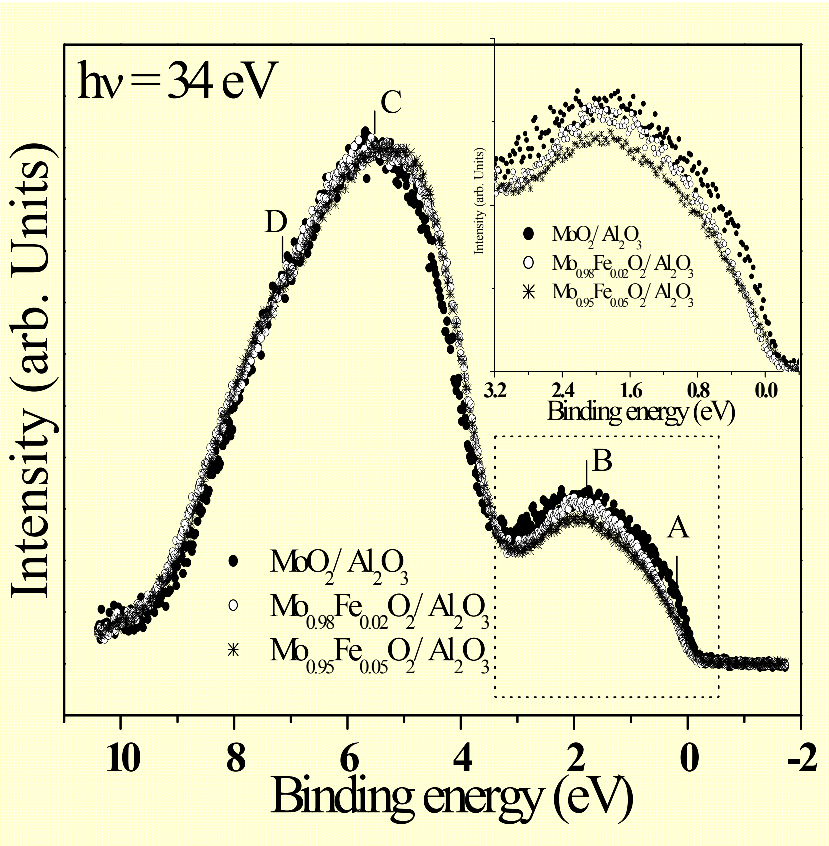
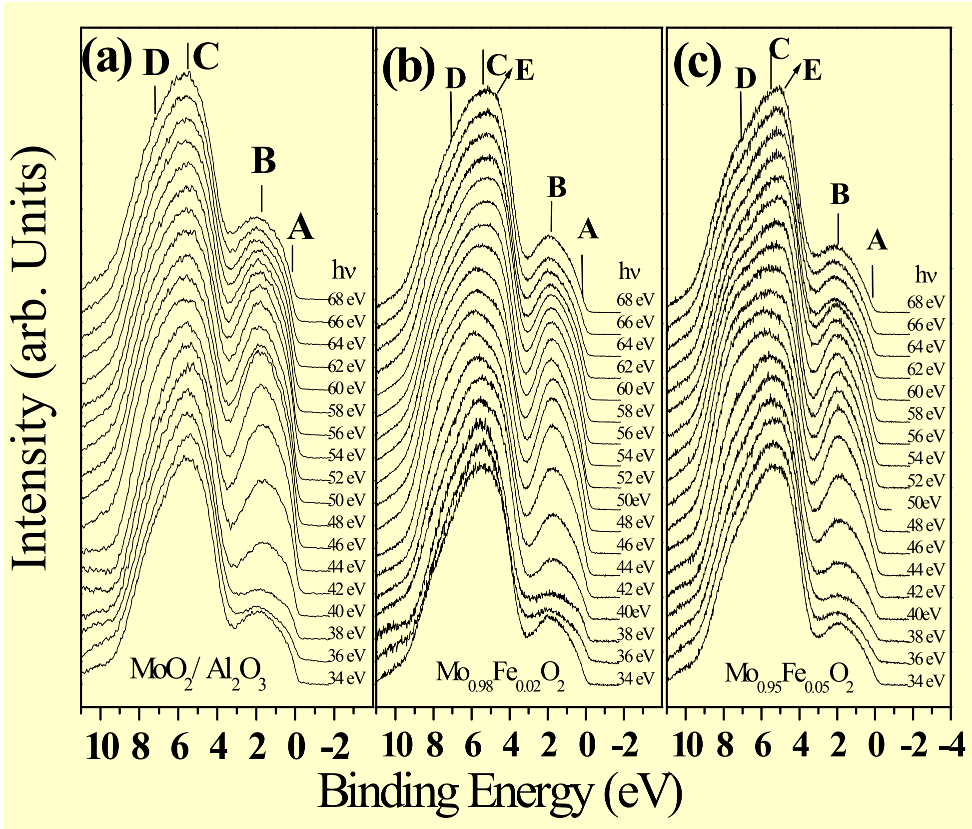
Abstract

The electronic structure of pulsed laser deposited $Mo_{1-x}Fe_xO_2$ (x=0-5at%) thin films has been investigated using resonant photoemission spectroscopy near the Mo4p absorption edge. In all the samples a broad resonance peak at ~46eV is observed in the whole area of the valence band, which indicates the contribution of the Mo4d states in the entire valence band region. The doping of Fe in these films leads to a decrease in Mo 4d states contributing to electronic states at lower binding energy region. In addition to this, we also observe a shoulder at 4.9 eV in the valence band spectra of doped samples. It is proposed that the origin of shoulder is due to the Fe hybridized states.



Valence band spectra of Fe doped and undoped Moly- oxide recorded at 34 eV photon energy.



Valence band spectra of (a) undoped and Fe doped moly oxide (b) and (c)*as a function* of photon energy from 34 to 68 eV.

JOURNAL OF APPLIED PHYSICS 104, 063717 (2008)

Possible origin of electronic phase separation in $La_{0.7}Ca_{0.3}MnO_3$

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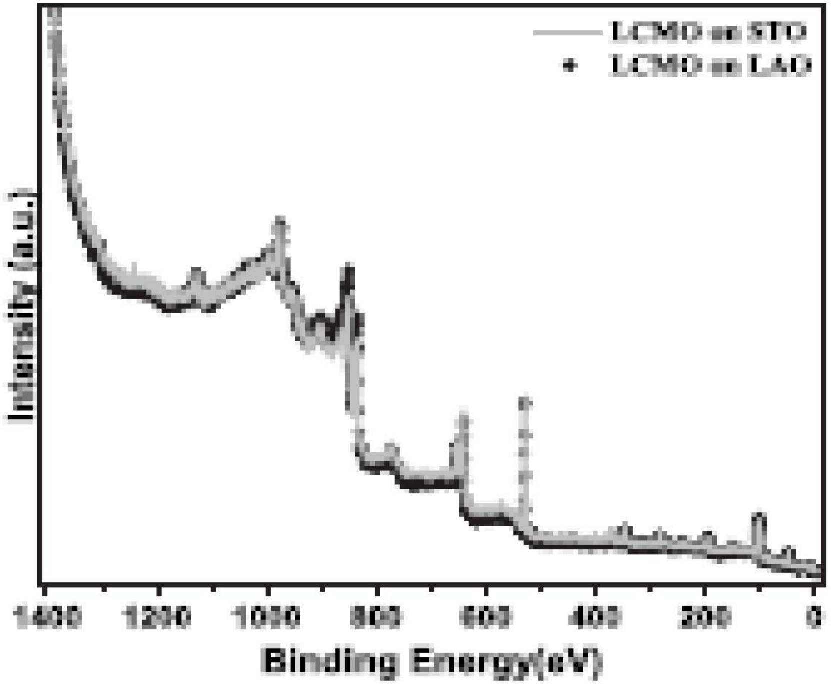
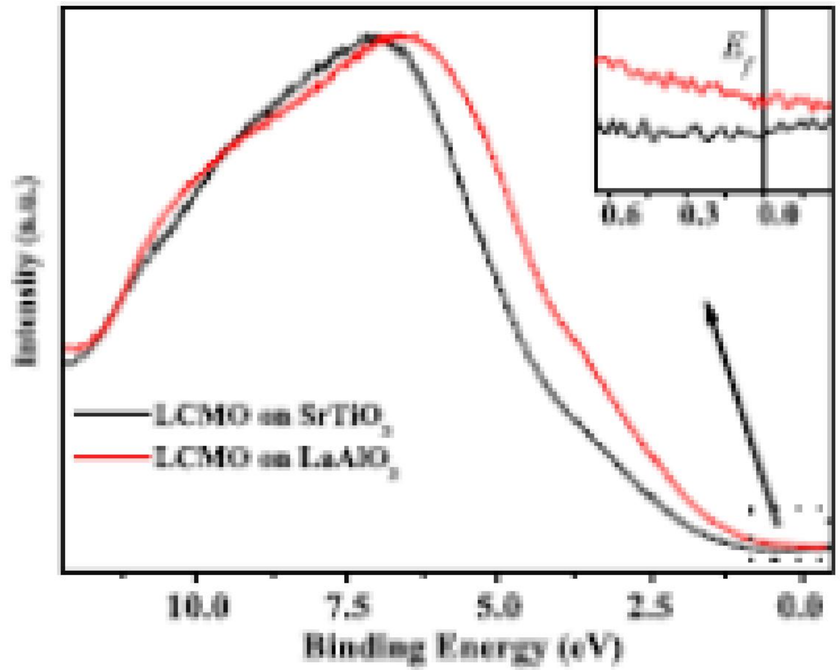
Abstract

The effect of substrate strain on the electronic valence band structure of LCMO thin films has been investigated. For this purpose LCMO thin films have been simultaneously grown on STO and LAO substrates using PLD technique. The chemical analysis of these samples is carried out by XPS and structural characterisation by XRD. Our results confirm that both the samples have same chemical composition but different strain configuration. The electronic structure of these samples is probed through valence band spectroscopy measurements on Indus-1 SR source. We observe that strain has a large effect on the valence band of LCMO. The results are explained on the basis of change in the crystal field splitting due to Mn-O bond length.





Core level XPS spectra for LCMO deposited on LAO (black filled circles) and STO (gray line) substrates.

Normalized valence band spectra for LCMO deposited on LAO (Red) and on STO (black) substrates; difference in the valence band spectra Is clearly visible. The inset shows the variation in DOS near the Fermi-level.

IOP PUBLISHING — JOURNAL OF PHYSICS D: APPLIED PHYSICS
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Effect of iron doping on electrical, electronic and magnetic properties of $La_{0.7}Sr_{0.3}MnO_3$

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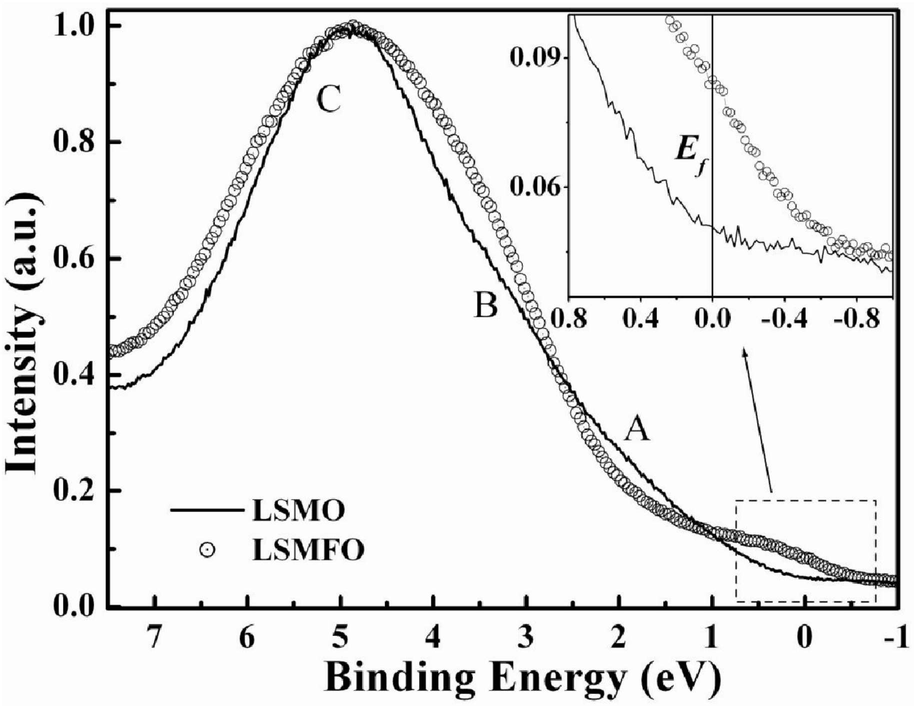
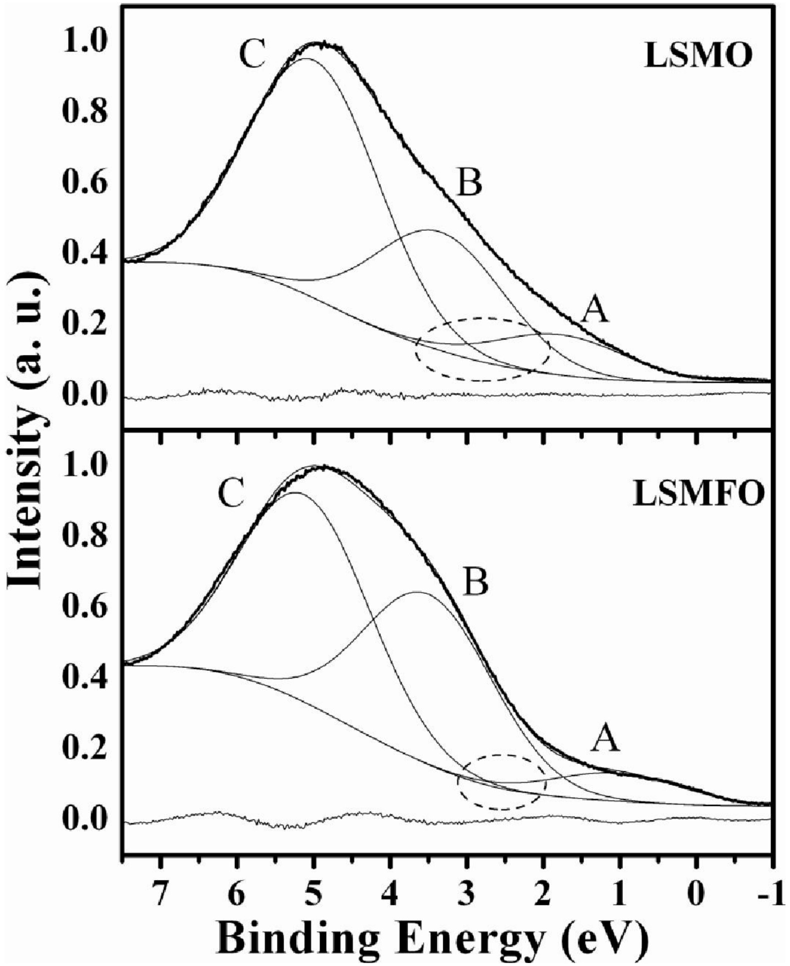
Abstract

The effect of 5 % Fe doping at Mn site, on the valence band structure of La0.7Sr0.3MnO3 has been investigated. Polycrystalline samples of La0.7Sr0.3MnO3 and La0.7Sr0.3Mn0.95Fe0.05O3 have been prepared by solid-state reaction route. The phase purity of these samples was confirmed using X-ray diffraction. The core level X-ray photoelectron spectroscopy measurements were performed to study the changes in the chemical composition. The valence band spectroscopy measurements on these samples, using the synchrotron radiation source, show a considerable change in the density of states at Fermi level with 5% Fe doping. The results are correlated with room temperature resistivity and magnetization data on these samples. These results suggest that though the density of states at the Fermi level increases on Fe doping, the conduction in LSMO gets hampered. This may be a result of changes in the hybridization of the orbitals due to Fe doping which modifies the MnO6 octahedra and hence the Mn3+-O-Mn4+ network.





The major changes that one can observe are that the e_g states are pushed towards E_F on Fe doping, thereby increasing the density of states at E_F

The decrease in the overlap of eg and O2p bands is shown with the dotted line.

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Iron substitution in CdSe nanoparticles: Magnetic and optical properties

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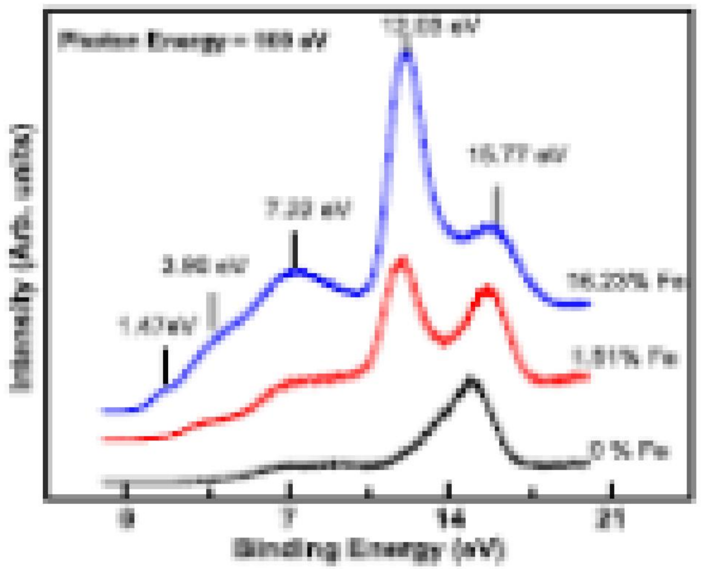
Abstract

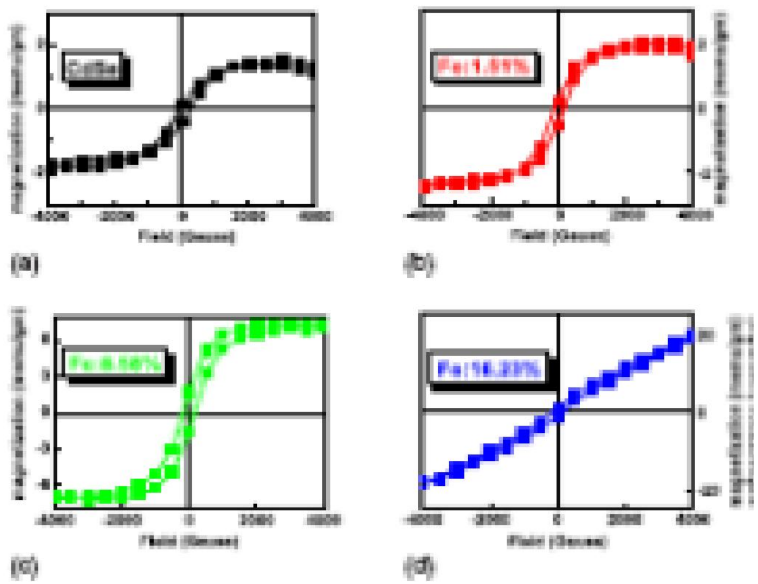
Chemically synthesized thiol capped undoped and Fe doped CdSe nanoparticles have been investigated using a variety of physicochemical techniques. The origin of room temperature ferromagnetism and significant changes in Ms value are discussed in terms of F-center exchange mechanism. (bound magnetic polaron).

Valence band spectroscopy



With the doping of iron in CdSe nanoparticles the valence band exhibits enhanced density of states near the E_f with distinct features at 1.47, 3.9 and 7.22 eV which are attributed to Fe3+ state of iron

Magnetization



Magnetization data shows change in saturation magnetization with respect to Fe doping percentage

JOURNAL OF APPLIED PHYSICS **108**, 103712 (2010)

Resonance photoemission studies of (111) oriented CeO_2 thin film grown on Si (100) substrate by pulsed laser deposition

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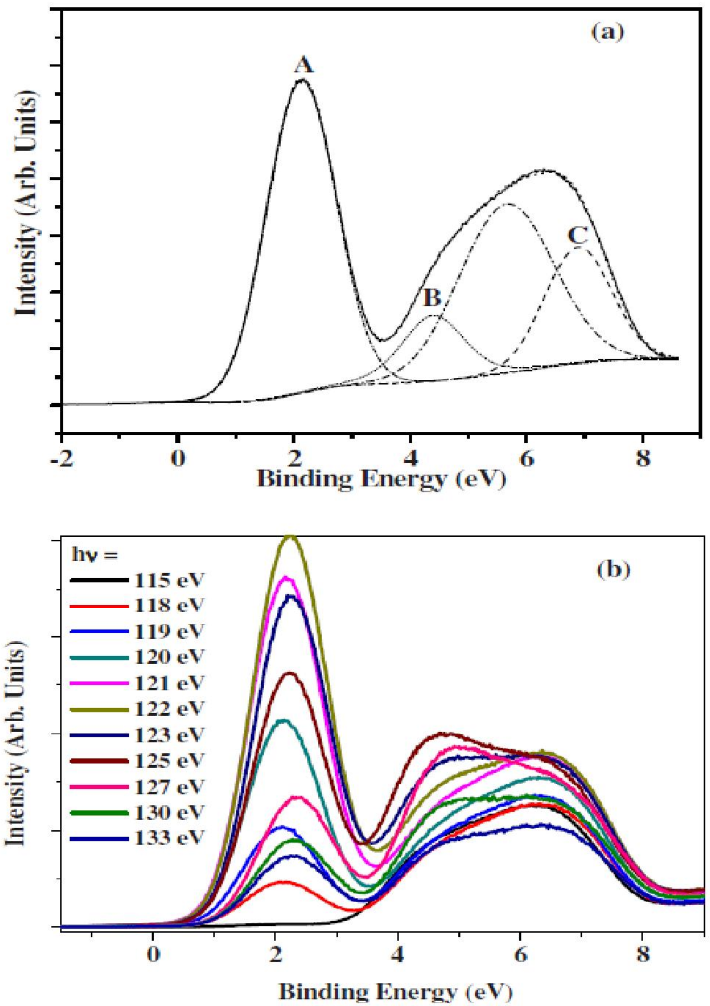
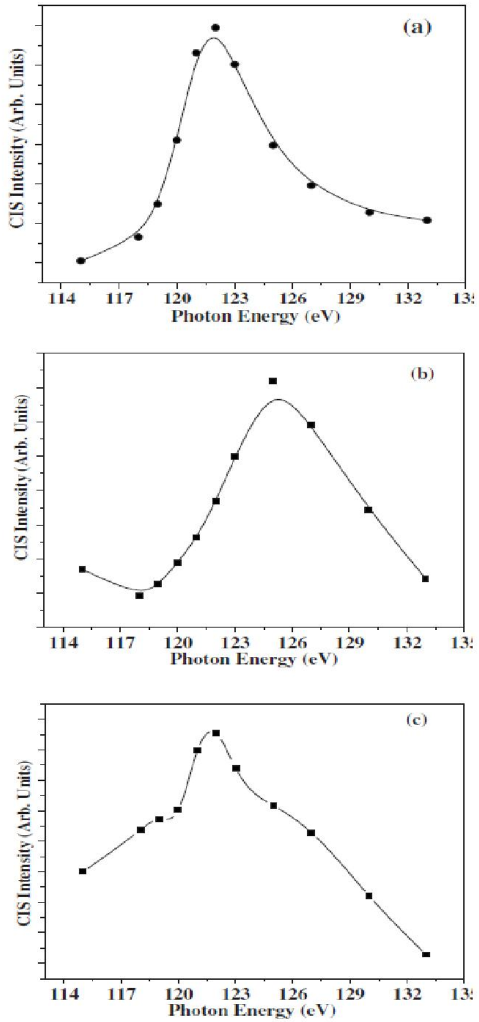
Abstract

The electronic structure of CeO2 thin film grown by pulsed laser deposition on Si 100 substrate has been investigated using resonance photoemission spectroscopy RPES. X-ray photoemission study on the film suggests that Ce has 3+ and 4+ valence states. Valence band spectra of the film show a feature at 2.1 eV of binding energy and a broad band at higher binding energy due to O 2*p derived* state. RPES measurements performed in the Ce 4*d*→*4f photoabsorption region show maximum* intensity for 2.1 eV feature at photon energy of 122 eV confirming it to be due to Ce3+ 4*f1 state.* RPES measurements also show maximum intensity for binding energy position of 4.4 eV in the broad band at photon energy of 125 eV, suggesting it to be due to Ce4+ 4*f0 state. Constant initial* state CIS versus photon energy plots also confirm these findings and suggest that the broad band is admixture of O 2*p and Ce 4f and 5d derived states.*



a) Fitted VBS for CeO2 thin film taken at PE of 120 eV. b) VBS recorded for CeO2 thin film at different photon energies .



a) CIS photoemission intensities of a feature A at binding energy 2.1 eV, b)feature B at binding energy 4.4 eV, and c)feature C at binding energy 6.7 eV .

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Electronic structure studies of Fe doped CeO_2 thin films by resonance photoemission spectroscopy

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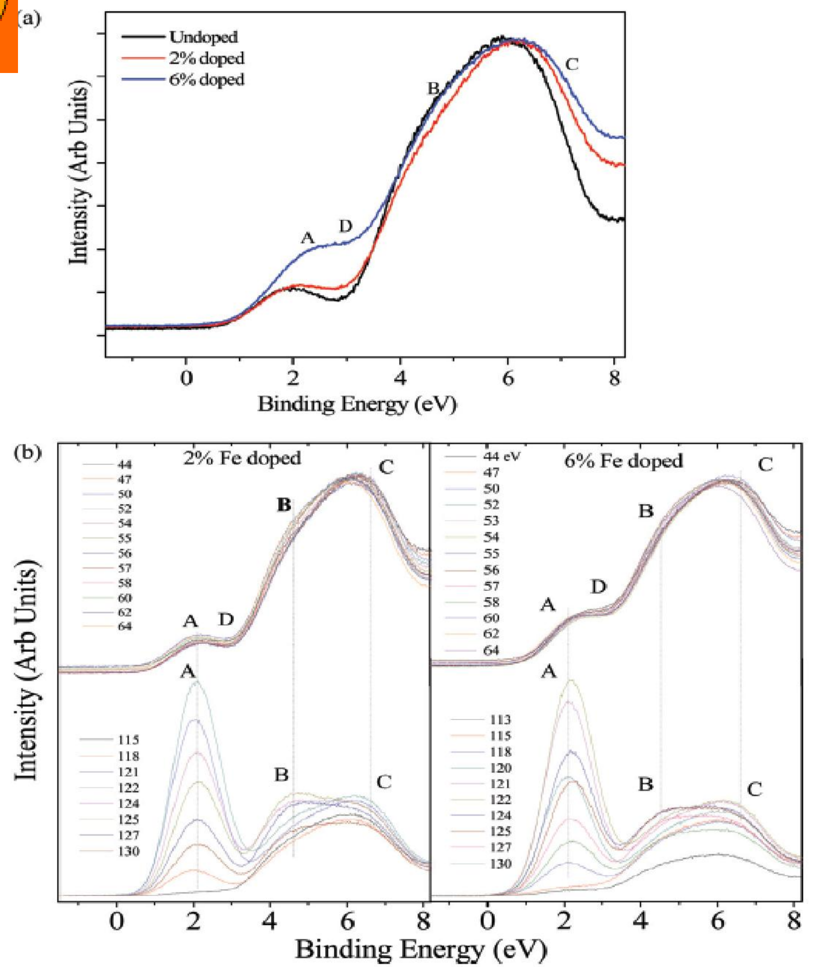
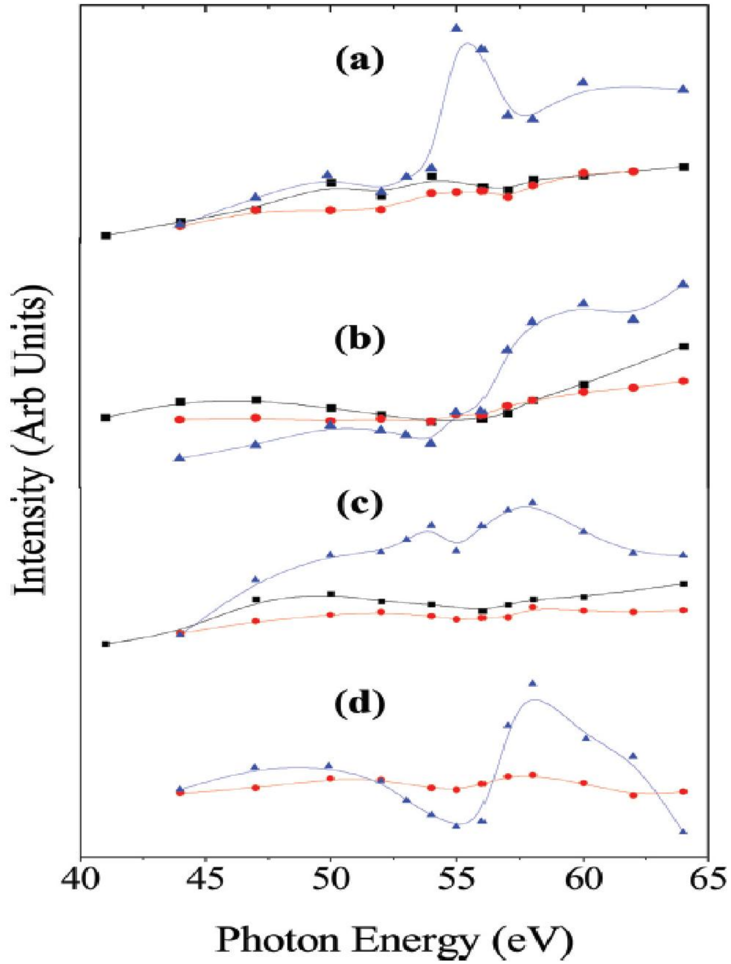
ABSTRACT

We have studied the modification in the electronic properties of pulsed laser deposited CeO2 thin films due to Fe doping (2 and 6 at %), with the help of x-ray photoemission spectroscopy (XPS) and resonance photoemission spectroscopy (RPES) measurements. XPS results indicate the ionic state of Fe in the Fe doped films, ruling out the possibility of Fe metallic clusters. Valence band spectra of CeO2 show an additional feature after Fe doping, suggesting its incorporation in the CeO2 matrix. RPES studies on these films reveal the hybridization between oxygen vacancy induced Ce localized states and Fe derived states.



(a) Valence band spectra of undoped, 2% and 6% Fe doped CeO2 thin films taken at photon energy of 50 eV, (b) Valence band spectra recorded for 2% and 6% Fe doped CeO2 thin films at different photon energy .



(a) CIS photoemission intensities as a function of photon energy for undoped (Black), 2% (Red) and 6% (Blue) Fe doped CeO2 thin film. (a) Feature A -2.1 eV), (b) Feature B -4.4 eV), (c) Feature C-6.7 eV) and (d) Feature D-2.6 eV.

Materials Letters 65 (2011) 2073–2075

Band offset in $Zn_{0.965}Cd_{0.035}O$/ZnO bilayer films

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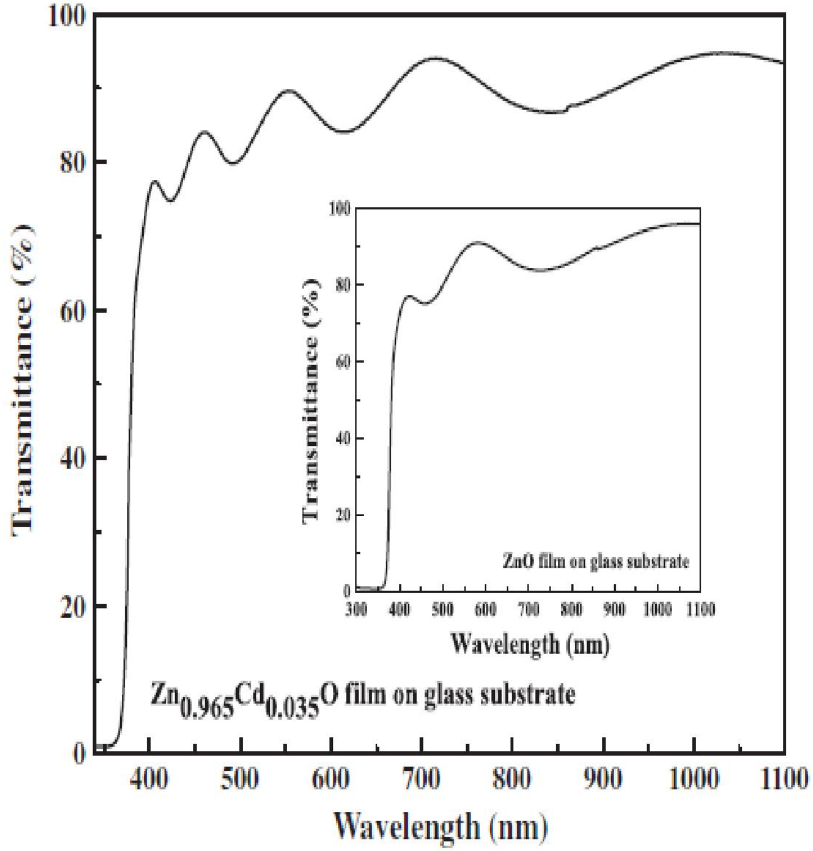
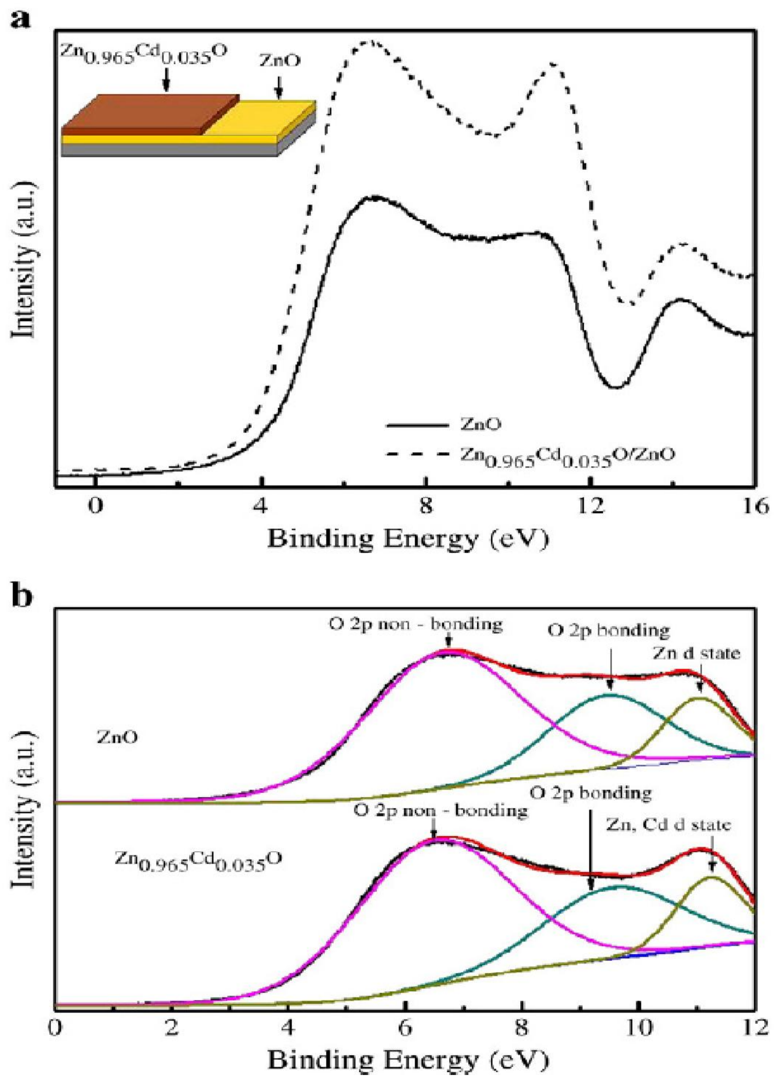
ABSTRACT

Zn0.965Cd0.035O/ZnO bilayer film has been developed using pulsed laser deposition (PLD) technique. The film is characterized by X-ray diffraction (XRD), energy dispersion analysis by X-ray (EDAX), UV-Vis and Valence band spectra (VBS). The XRD pattern confirms the single phase crystalline nature of the deposited film. The UV-Vis spectra establish a reduction of band gap (≈340 meV) in the ternary alloy film of Zn0.965Cd0.035O/ZnO. The VBS shows shift in the peak corresponding to nonbonding oxygen p states. We also obtained valence band offset of 191 meV in the film showing the rise of valence band. The calculated conduction band offset is found to be −51 meV which confirms the lowering of the conduction band in the ternary alloy film.



UV-Vis spectrum of Zn0.965Cd0.035O film on glass substrate. The inset shows the UV-Vis spectrum of ZnO film on glass substrate.



a)VBS of bilayer Zn0.965Cd0.035O/ZnO film deposited on Si (100) substrate. The inset shows the surface layers of the film from which the spectra is taken.(b) Deconvolution of the VBS spectrum obtained from ZnO and

Zn0.965Cd0.035O surface layers.

APPLIED PHYSICS LETTERS **101**, 242402 (2012)

CrossMark

Resonant photoemission study of epitaxial $La_{0.7}Sr_{0.3}MnO_3$ thin film across Curie temperature

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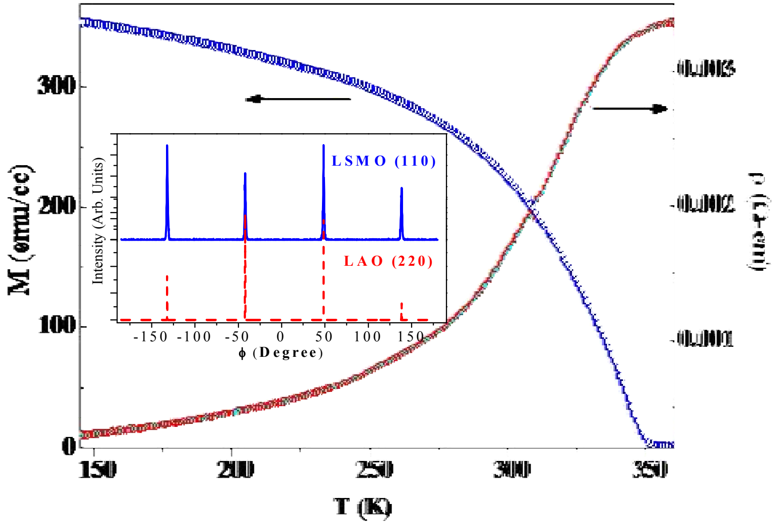
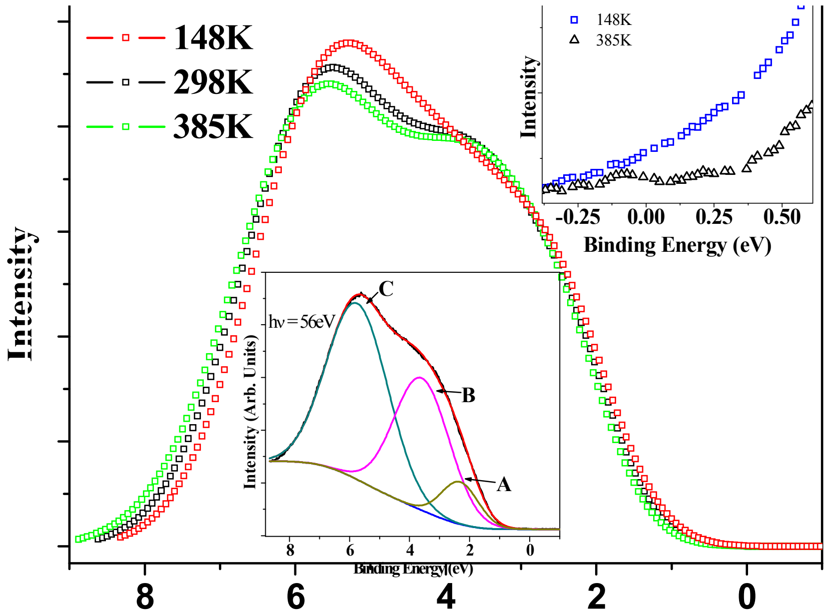
ABSTRACT

The electronic structure of epitaxial La0.7Sr0.3MnO3 thin film grown on LaAlO3 (001) substrate has been studied by resonant photoemission spectroscopy across Curie temperature (TC). Temperature dependent variations in the valence band structure divulge that beyond T_C, Mn-O 2p hybridization is reduced. It is found that nature of the states in the lower binding energy range above T_C would be rather due to Mn-3þ ions, whereas below T_C Mn-3þ and Mn-4þ contributes equally in the low binding energy region. At higher binding energy values, Mn-4þ contribution is larger in the Mn-O hybridization at all temperatures.

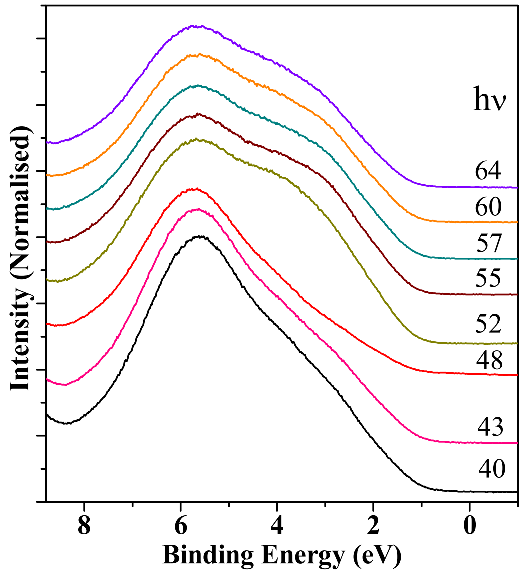


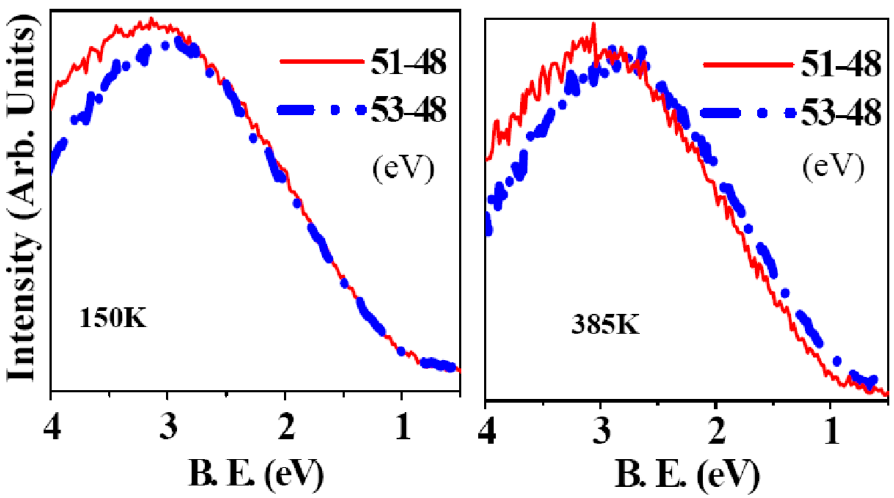


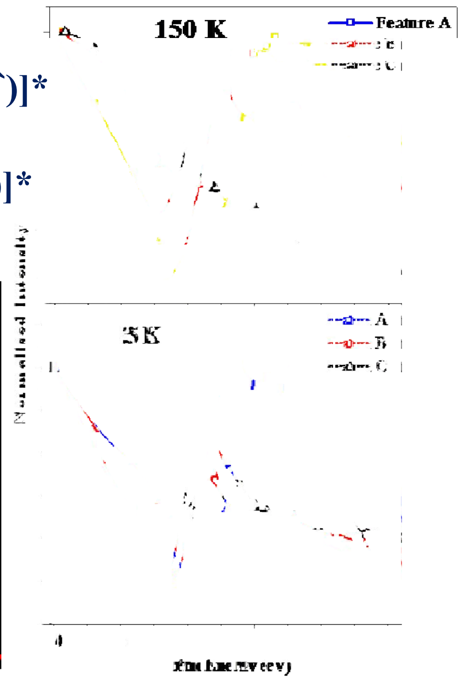
$Mn3p^6 3d^3(t_{2g}{}^3\uparrow) + h\upsilon \rightarrow Mn[3p^5 3d^4(t_{2g}{}^3\uparrow eg_1{}^1\uparrow)]^*$
$\rightarrow Mn3p^6 3d^2(t_{2g}{}^2\uparrow) + e^-$ (for Mn^{4+})

$Mn3p^6 3d^4(t_{2g}{}^3\uparrow eg_1{}^1\uparrow) + h\upsilon \rightarrow Mn[3p^5 3d^5(t_{2g}{}^3\uparrow eg_1{}^1\uparrow eg_2{}^1\uparrow)]^*$
$\rightarrow Mn3p^6 3d^3(t_{2g}{}^3\uparrow) + e^-$ (for Mn^{3+})

$Mn3p^6 3d^4(t_{2g}{}^3\uparrow eg_1{}^1\uparrow) + h\upsilon \rightarrow M[3p^5 3d^5(t_{2g}{}^3\uparrow eg_1{}^1\uparrow t_{2g}{}^1\downarrow)]^*$
$\rightarrow Mn3p^6 3d^3(t_{2g}{}^3\uparrow) + e^-$ (for Mn^{3+})







Conclusion

➢ **In general x-ray absorption spectroscopy and valence band spectroscopy techniques are complementary to each other. So using soft x-ray beamlines on Indus-1 and 2 one can effectively probe the electronic properties.**

Let's try soft X-ray absorption!!

THANK YOU

for your kind attention

☺