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T.2 : Research activities using the synchrotron radiation source : Indus-1

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This article reviews the various science and technology experiments performed on Indus-1 over the last four years. The objective of this article is to highlight the scope of Indus-1 synchrotron facility with few specific examples, and to encourage more users to come forward in planning experiments with Indus-1.

Indus-1, a 450 MeV electron storage ring, is a good source in soft x-ray / vacuum ultra violet (40 to 1000 Å) and infrared regions of the electromagnetic radiation spectrum. The critical wavelength of the emitted synchrotron radiation is 61 Å. The electron beam completes the closed orbit path using four 1.5 T bending magnets (BM) with a bending radius of 1 m. Each BM vacuum chamber has two synchrotron radiation ports. Fig.T.2.1 gives the schematic representation of Indus-1 storage ring along with location of various beamlines. These beamlines have been installed on three BM ports (DP1, DP2, and DP3). At present, on Indus-1, five beamlines are operational. On DP1, high resolution vacuum ultra violet (VUV) spectroscopy beamline and photo electron spectroscopy (PES) beamline; on DP2, angle resolved photo electron spectroscopy (ARPES) beamline and soft x-ray-VUV(SXUV) reflectivity beamline; and on DP3, photo physics beamline are installed. Efforts are underway to install an infra red beamline on DP3. Front end of SXUV reflectivity beamline on DP2 is being modified to accommodate a photo absorption spectroscopy (PAS) beamline. Characteristics of the various beamlines operational on Indus-1 are given in Table-1. Details of the Indus-1 and various beamlines are available in *Current Science* as a special section: "Indus-1 synchrotron" [1] and in Indus-1 Activity Report 2003 [2].

Soft x-ray–VUV reflectivity beamline

The soft x-ray-VUV (SXUV) reflectivity beamline has reflectometer and time of flight mass spectrometer as experimental stations. The reflectometer station is used for the investigation of optical response of various materials with emphasis on measurements near the absorption edges, where experimentally measured response is scarce. The focus is to undertake interface studies in thin films structures and for testing the performance of the soft x-ray optics at the designed wavelength. The reflectometer is equipped with two axis high vacuum compatible goniometer operating at ~10^{-7} mbar vacuum [3]. The design and fabrication of optical devices in x-ray region require reliable knowledge of the soft x-ray optical response of the materials. The penetration of soft x-ray is small (<1 micron), and any contamination on the surface can significantly influence the optical properties. The optical properties in 80-200 Å regions were measured for the two sides of float glass substrate [4,5]. The optical properties of the two sides were significantly different, due to diffusion of iron and tin on one surface, during the production process [6].

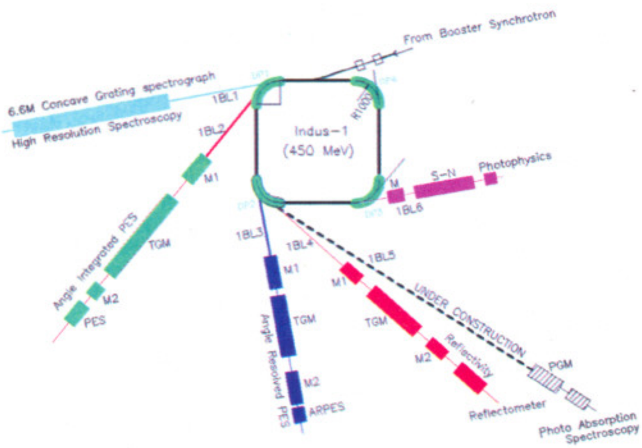

Fig.T.2.1: Schematic representation of experimental hall of Indus-1 along with location of various beamlines

In the soft x-ray region, for some elements, near the absorption edges, the real part of refractive index shows a sign reversal. This sign reversal was measured around boron K absorption edge (67 Å), by studying soft x-ray reflectivity of boron carbide thin films [7,8] . The photon energies can be tuned, where the refractive index of the material is very close to the refractive index of vacuum and interaction with the material becomes negligible. This can be used for structural studies of embedded layers. This was demonstrated by studies of iron (180 Å)-B_4C (600 Å) thin film structure deposited on float glass, using soft x-ray reflectivity measurements near boron K edge. Using reflectivity measurements at 66.9 Å, where the real part of boron refractive index approaches zero, morphology of iron layer was probed with high sensitivity, as the interaction of radiation with top B_4C layer is negligible [7, 8].

X-ray multilayers are important optical elements for soft and hard x-ray region of the electromagnetic spectrum. These structures are used for near normal incidence optics in soft x-ray region and for enhancing the grazing incidence reflectance in hard x-ray region. The period length of these structures is in the range of 15 to 70 Å. A research program is underway at RRCAT, on the development of x-ray multilayer optics. The reflectivity of Mo/Si, W/C, Pt/C x-ray multilayer structures was measured [5,7,9]. To test the performance of W/C x-ray multilayers under high heat load, soft x-ray reflectivity studies at 80 Å were performed with *in-situ* annealing of multilayers up to 400 °C. The period expansion and smoothening of interfaces with annealing temperature were observed [5, 11]

In the plane of the electron orbit, the synchrotron beam is linearly polarized and becomes elliptically polarized as one move above or below the electron orbit. One needs to monitor the polarization state of the synchrotron beam before and after it interacts with the sample. For SXUV beamline, a polarimeter, working in 200 to 1000 Å region, was designed [12,13]. For the measurement of polarization in 125 to 150 Å region, Mo/Si multilayers with high reflectivity at 45° were evaluated [14].

Silicon nitride films are of great technological importance due to their novel physical properties. Its high dielectric constant and good diffusion barrier characteristics have made them more attractive for modern integrated semiconductor devices. Combination of optical and x-ray transparency along with stiffness of silicon nitride films allows one to fabricate thin membranes for x-ray masks. These membranes are exposed to intense x-ray radiation in the soft x-ray / VUV region. The response of these films under soft x-ray / VUV illumination is not properly understood. Photo-illumination experiments using photons up to 250 eV from Indus-1 and *in-situ* soft x-ray reflectivity experiments were performed to analyze subsequent changes. The major finding was that on illumination with soft x-ray photons, there is breakage of hydrogen bonds and subsequent out diffusion of hydrogen, which leads to compaction of silicon nitride network [15].

ASTROSAT is one of the most ambitious space astronomy telescope programme, initiated by space science community in India. Amongst its several payloads, a Soft X-ray imaging Telescope (SXT), sensitive to 0.3 to 8 keV, is planned. It will use thin shells of x-ray reflecting surface of gold, adhering to aluminum foils. The soft x-ray reflectivity performance was tested on SXUV reflectivity beamline [16]. Reflectivity around 300 eV falls significantly due to a thin contamination layer above the surface of gold.

An experimental station for the investigation of photo-ionization processes in molecules has been in operation at the SXUV reflectivity beamline for the last four years. The purpose of this project is to unravel the dissociative ionization mechanisms in molecular ions. The focus is on multiply charged molecular ions, which owing to their instability are not very well studied. Photons are ideal agents for bringing out multiple ionization, as they can selectively remove inner

Beamline	Range Post	Beamline optics		λ/Δλ	Experimental station
		Pre and mirror	Monochromator		
SXUV Reflectivity	40-1000 Å	Au coated Toroidal	1.4 meter TGM with three inter changeable gratings	~400	Reflectometer and time of flight mass spectrometer
PES	60-1600 Å	Pt coated Toroidal	2.6 meter TGM with three inter changeable gratings	~600	EA 125 1800 hemisperical analyzer
ARPES	40-1000 Å	Pt coated Toroidal	1.4 meter TGM with three inter changeable gratings	~400	Angle resolved AR-65 electron analyzer (±1o)
High resolution VUV	700-2000 Å	Au coated cylindrical	6.65 meter off plane Eagle mount spectrometer	~70000	Absorption cell and high temperature furnace
Photo Physics	500-2500 Å	Au coated Toroidal	1 meter Seya-Nomioka	~1000	Absorption cell and UHV chamber with sample manipulator.

Table-1: Characteristics of beamlines operational on Indus-1

or outer shell electrons. The technique used is a combination of ion time-of-flight spectrometry and momentum spectroscopy. The technique becomes far more powerful, when multiple fragments arising from the break-up of a single molecular ion are detected. Kinematically complete measurements can thus be made and changes in the structure and energy levels of unstable molecular ions can be discovered. In large molecules, dissociation may take place step by step over a time-scale as short as a picosecond. Multi-ion coincidence technique enables us to identify such fast processes. An experimental station was first set up by scientists from PRL, Ahmedabad in collaboration with X-ray Optics Group at RRCAT, and the project was strengthened by the coming together of a team from IIT-Madras. Experiments so far have investigated double and triple ionisation of Ar [17], CO_2[18], CO[19], and SF_6[20]. The highlights of the investigations are the observation of bent dissociative states in $CO2^{3}+$ and the formation of F_2^+ from SF_6.

Photoelectron spectroscopy beamline

This photo-electron spectroscopy (PES) beamline is developed by UGC-DAE-Consortium for Scientific Research, Indore. The experimental station on PES beamline comprises of EA125 1800 hemispherical analyzer, and a sample preparation chamber. The experimental station has an argon ion gun for surface cleaning of thin films, a diamond file to scrap the pallet samples, sample heating (up to 450 ^{0}C) and sample cooling (down to -160 ^{0}C), magnetic sample transfer mechanism to transfer sample from preparation chamber to measurement chamber without breaking the vacuum and residual gas analyzer to check quality of vacuum. The beamline is used for the determination of electronic structure in a wide verity of materials.

The effect of substrate strain on the electronic valence band structure of $La_{0.7}Ca_{0.3}MnO_3$ thin films was investigated. Strain in these films significantly modifies the valence band of $La_{0.7}Ca_{0.3}MnO_3$. The results are explained on the basis of change in the crystal field splitting due to Mn-O bond length. Electronic structure of iron doped cobalt MoO_2 thin films were studied using resonant photoemission near Mo 4p [21]. The doping of Fe in these films leads to a decrease in Mo 4d states contributing to electronic states at lower binding energy region.

The surface photo voltage (SPV) and the charging effects modify the PES spectra of depleted semiconductors. These effects are reduced to negligible values in the presence of excess plasma (due to absorption from a secondary white light source) density of ~ 10^{18} cm^3/sec. The effect of the charging and SPV is very small on the value of the valence bands offset measured in the presence of excess plasma. A new method to measure the valence band offset of *ex situ* prepared samples was investigated by measuring the valance band spectra in presence of secondary light source. This method to measure the valence bands offset is useful for samples prepared in *ex situ* conditions and with film thickness of the order of 10 to 100 nm [22]. The technique has been applied for the measurement of nano-porous GaP [23] and n-type GaP [24]

Electronic structure of deposited and annealed Ti/Ni, Fe/Al, Fe/Si and Si/Ge multilayer structures was investigated using the PES beamline. FeAl phase formation in Fe/Al multilayers was studied using valance band photo electron spectroscopy and an attempt was made to correlate the electronic structure with magnetic and transport properties [25-27]. Valance band of iron silicide phase formed at iron silicon interface was investigated [13,28] and the measurements were correlated with other structural parameters obtained by x-ray diffraction, hard x-ray reflectivity and cross sectional transmission electron microscopy. TiNi phase formation in Ti/Ni multilayers was studied using valance band photoemission and photo resonance studies [29-31]. With an aim to generate materials with variable band gap structures, electronic band structure in Si/Ge multilayer structures were investigated as a function of micro structural parameters [32-33]. Electronic structure of Co and Co/semiconductor interface were studied and correlated with magnetic and transport properties [34].

Angle Resolved PES Beamline

The angle resolved photo-electron spectroscopy (ARPES) beamline is developed by Spectroscopy Division, BARC. The ARPES station on this beamline has recently been upgraded and consists of an angle resolved AR-65 (Omicron) electron analyzer ($\pm 1^{\circ}$), sample manipulator with facilities for heating the sample up to 1000°C or cooling down to liquid N_2 temperature. An *in-situ* sample load lock facility integrated with the experimental station allows the loading and unloading of samples into the spectrometer chamber without venting. The maximum photon energy available for excitation is 310 eV. The beam spot is around 1 mm (vertical) x 2 mm (horizontal) at the sample position and the maximum sample size which can that be accommodated on the sample holder is 20 mm diameter and 0.5 mm thickness. The type of the samples on which PES studies can be performed using this beamline, are non-volatile solid samples. The energy resolution of the spectrometer is ~20 meV at room temperature. Test runs on upgrade station have been performed. Presently PES beamline is being used to record and investigate valence band and core level spectra of lead oxide samples.

Photo-physics Beamline

The photo-physics beamline is developed by Spectroscopy Division, BARC. It is a medium resolution facility, which consists of a 1meter Seya-Namioka monochromator for wavelength selection in the range of 500 – 3000 Å. Using a 2400 g/mm ion etched laminar grating as the dispersing optical element, the resolution achieved is about 1.5Å. The existing experimental station enables gas phase studies in the region 1100 – 3000 Å. A sample chamber with a sample manipulator capable of holding samples of 2.5 cm diameter can be used to perform transmission / reflection experiments on solid samples. Using the photo-physics beamline and the experimental facilities, gas phase UV-VUV absorption spectroscopy investigations on methyl iodide, ammonia, carbon disulphide, difluoromethane, benzene, and reflection experiments on gadolinia, silica thin films have been carried out [35,36].

Radiation physics experiments using Indus-1

Electron accelerators in Indus-1 are used for radiation physics related research [37-42]. The major work carried out in this area was in the investigation of electro-magnetic cascade generation in various material mediums and deducing the shower parameters like maximum absorbed dose, shower maximum etc. This work has relevance in source term determination, high energy accelerator shielding and in radiation damage. Investigation of absorbed dose build up factors on account of the shower generation was experimentally studied. Experimental measurement of the shower propagation in water phantoms leads to the deduction of high energy correction factors for various radiation detectors used in personnel protection.

Conclusion

We have tried to demonstrate here the usefulness of Indus-1, in generating good science and technological input to some of our ongoing research and development programs. We are confident that with active research interest of many more research groups, we should be able to explore many more applications. The utilization potential of Indus-1 is immense, and the need of the hour is to have more number of users coming forward to use this machine, and put academic pressure on the machine builders to improve the capability of the machine to its limit, and to provide beam of even better quality. The SR machine group and the users have to work hand in hand, to make this possible.

As Indus-1 is a low energy machine, the higher order energy contamination is low. Heat loads and radiation safety problems are insignificant. It is the first SR source in the Indian sub-continent, emitting in vacuum ultra violet / soft x-ray region. This region of the electromagnetic spectrum is very difficult to use, but has significant science and technology application. Most of the low energy storage rings (<500 MeV) have been substituted with high energy third generation machines with state of art insertion devices. On the new generation machines, due to limited flexibility, it is sometimes difficult to design experiments to test a scientific idea. Indus-1 can serve as a test bed in designing novel experiments, and well defined scientific ideas can be further explored using the new generation SR facilities.

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