

## AWARENESS WORKSHOP ON LOW TEMPERATURE AND HIGH MAGNETIC FIELD FACILITIES AT UGC-DAE CSR, INDORE

An awareness workshop under the aegis of DST on “Low Temperature and High Magnetic Field (LTHM) Facilities at UGC-DAE CSR, Indore” was held during 3-4 July, 2006. To promote research in the area of low temperature and high magnetic field, DST has sponsored two systems for CSR, Indore. One of them is a 14T/0.3K system for heat capacity and resistivity and the other is 14T/2K VSM where in magnetization can be measured down to 2K in fields up to 140 kOe. Recently these two national facilities became operational and made available to the researchers. The aim of this workshop was to generate awareness about these powerful facilities and engage the young researchers from across the country by exposing them to various topics in this area. About 100 participants attended the workshop, out of which 25 were outstation participants. These research scholars and young faculty members from different universities/institutes had a chance to interact with a large number of experts numbering about 15 through formal lectures and informal discussions.



After the formal welcome by Prof. Ajay Gupta, Prof. R. Srinivasan inaugurated the workshop. Dr. P. Chaddah presented overview of the LTHM capabilities of the consortium. He briefly mentioned about the initiative taken by committee under the chairmanship of Prof. R. Srinivasan to setup LTHM national facilities which was followed up by the then Director of CSR, Dr. B. A. Dasannacharya submitting a project proposal on behalf of this consortium. There was brief mention of cryogenic and low temperature facilities of the consortium, their utilization by users, research carried out in different groups and justification for the need of high fields for different systems of current interest giving examples of field induced 1<sup>st</sup> order transitions in vortex matter, intermetallics, manganites etc.

Prof. A. K. Raychaudhuri after giving a brief comment on the methodology for low temperature physics discussed about coupling unusual probes to low temperatures. He discussed in details about noise as a probe for condensed matter and produced a proof of suppression of universal conductivity fluctuation by magnetic field. Prof. D. D. Sarma talked about the magnetism and magneto-transport of Fe-based double perovskites and correlated it with the band structure calculation. Based

on their experimental and theoretical studies a model is proposed to explain the origin of the robust ferromagnetism in double perovskite,  $\text{Sr}_2\text{FeMoO}_6$ .

Dr. V. Ganesan described the 14 T/0.3 K system for measurement of specific heat and resistivity. He discussed the drastic effect of magnetic field on specific heat and resistivity of different systems showing diverse phenomena. Some representative results on different systems like shape memory alloys, heavy fermion systems and superconductors were discussed. Dr. Alok Banerjee discussed about the limitations and capabilities of the 14 Tesla Vibrating Sample Magnetometer (VSM) and compared it with a lower field (7 Tesla) homemade system. He showed that with the use of this 14-Tesla VSM they could unambiguously conclude a ferromagnetic ground state for minimally destabilized narrow band robust charge ordered system.

Prof. A. K. Nigam started his talk giving an outline of various high magnetic field facilities existing in the country and elsewhere. He discussed the need of high magnetic field to probe various anomalies observed in different inter-metallic and heavy fermion systems giving examples from their work. Dr. S. B. Roy discussed about meta-magnetic materials; disorder broadened first-order transitions giving rise to field-temperature induced functional properties and phase coexistence in such systems. He gave examples from their studies on various systems to prove the existence of kinetic arrest of the first-order transformation process at low temperature which has drastic effect on low temperature properties and masks the ground state of the system.

Prof. A. K. Majumdar talked about extraordinary Hall Effect in self-assembled Ni- nanocrystallites embedded in TiN matrix. He showed how this system can be used for high density recording without entering the 'superparamagnetic limit'. From detailed high field magnetization, resistivity and Hall Effect studies it is shown that in the nano-scale regime, though the magnetic behaviors are drastically modified, the usual scaling law for the extraordinary Hall Effect still remains valid. Prof G. Baskaran gave a talk on the novel electric field effect on the Landau levels of Graphene. He showed how simple it was to make 2-D graphine sample and measurement of Hall effect reveals rich physics. Interesting theoretical ideas are put forward to explain the experimental observations.

Prof. E. V. Sampathkumaran discussed about the magnetism in the spin-chain compounds. He showed unusual glassy features in different systems with interesting magnetic properties; with some similarities as well as contrast and different sensitivity to the magnetic field. It was shown that these materials can be a good playground to study dimensional crossover, Jahn-Teller effect, disorder, geometrical frustration etc. Dr. Rajeev Rawat described the indigenously developed resistivity and specific heat setup in the commercial 8-Tesla Oxford cryostat. He presented resistivity, magneto-resistance and in-field specific heat results on diverse systems including single crystals, polycrystals and quasicrystals.

In the final session, the participants gave their feedback and the concerned scientists from the consortium responded specific queries about the LTHM facilities. Subject experts including Dr. B. A. Dasannacharya, Dr. T. S. Radhakrishnan, Prof. B. K. Srivastava gave their valuable comments. The concluding remarks were given by Prof. R. Srinivasan. He commended the effort by the consortium to sustain the steady growth in the LTHM facilities and ever increasing user base. He stressed the need of developmental activities along with the use of commercial setups to maintain a high level of instrumentation to generate high quality science.



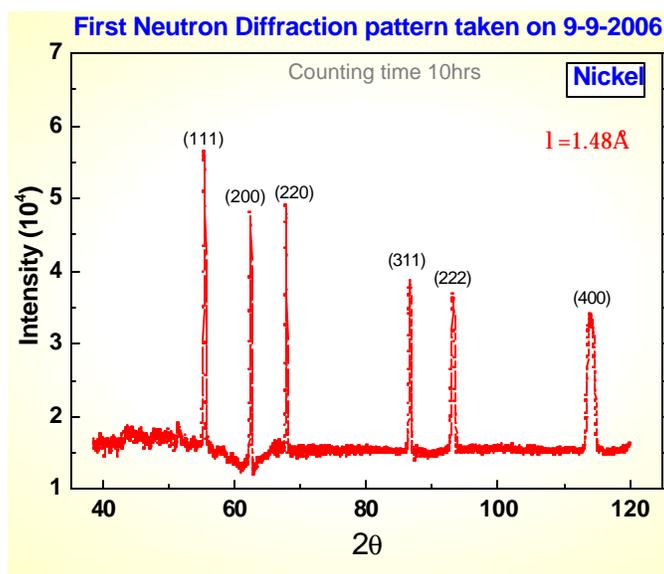
14 Tesla / 0.3 K System



## New developments in research facilities at UGC-DAE CSR Mumbai centre

### Focusing Crystal based Neutron Powder Diffractometer

The neutron powder diffractometer on multi-instrument tandem neutron beam line at TT1015 beam port, Dhruva Reactor, developed by Mumbai Centre of the Consortium with active participation from university scientists has been commissioned. The first neutron powder diffraction pattern was recorded on nickel powder on September 9, 2006 as shown in figure. This is a preliminary data with rough background correction and the angles indicated on the xaxis are approximate. This diffractometer employs open beam geometry and doubly focusing (horizontal and vertical directions) silicon monochromator and a number of position sensitive detectors. The silicon monochromator was set to reflect 1.48 $\text{\AA}$  neutrons from the (511) plane. It is possible to obtain other wavelengths, namely, 1.17  $\text{\AA}$  and 2.30  $\text{\AA}$  from (533) and (311) planes, respectively, by a simple rotation of the monochromator, and 1.76  $\text{\AA}$  from (331) plane can be obtained by turning the monochromator upside down. With monochromator take-off angle of 90 $^\circ$ , the diffractometer is designed to have high resolution that is nearly constant over wide scattering angles of 5 $^\circ$  to 120 $^\circ$ . Improvements/refinements to the instrument are being carried out by putting a nose cone, appropriate slit arrangement, oscillating radial collimator, etc, to enhance the signal to noise ratio, reduce background, etc. This instrument is expected to be available for users very soon.



### Foreign visits by Faculty and Students of CSR

Name	Designation	Period	Place
Dr. M. Gupta, Mumbai	Scientist	06 - 30 Nov. 2006	ILL France and PSI Switzerland
Mr. S. Chakravarty, Indore	Research Scholar	06 July – 31 Aug. 2006	ESR, Grenoble, France and TU Clausthal, Germany
Prof. A. Gupta, Indore	Centre-Director	06 – 24 July 2006	ESR, Grenoble, France
		July - August, 2006	Gordon Research Conference, Plymouth, USA & other universities in USA
		25 Mar. – 01 Apr. 2006	Elettra, Trieste, Italy
Mr. A. Shukla, Indore	Research Scholar	29 may – 14 Jul. 2006	FHI & BESSY-II, Berlin, Germany
Dr. S. S. Ghugre, Kolkata	Scientist	15 Apr. – 17 May 2006	Univ. Notre dame, Indiana, US
Mr. R. Brajpuria, Indore	Research Scholar	08 – 29 May 2006	ICTP, Trieste, Italy
Mr. A. Sharma, Indore	Research Scholar	08 – 29 May 2006	ICTP, Trieste, Italy
Ms. P. Rajput, Indore	Research Scholar	25 Mar. – 01 Apr. 2006	Elettra, Trieste, Italy
Dr. V. G. Sathe, Indore	Scientist	21 Mar. – 20 Jun. 2006	Elettra, Trieste, Italy
Dr. A. Saha, Kolkata	Scientist	17 – 21 Sept. 2006	Asia Pac. Symp.Rad. Chem., Shanghai, China
Ms. S. Tripathi, Indore	Research Scholar	30 Jul. – 02 Aug. 2006	Int. Conf. Syn.Rad. Mat. Sc. , Chicago, US

## Magnetron sputtering system for deposition of thin films and multilayers

Any method normally used for physical vapor deposition of thin film can be adopted for multilayer structure fabrication. The most commonly used techniques are sputtering and electron-beam evaporation. The choice mainly depends on specific goals and types of materials used for evaporation. Recently we have designed and installed a versatile DC/RF magnetron sputtering system at CSR, Indore. The main motivation behind developing the present system is to fabricate good quality thin films and multilayer structures of metals and compounds. This sputtering system consists of two DC and one RF sputtering source for deposition of metallic and insulating thin films and multilayer structures with substrate heating arrangement. The specification for the system sources are given below:

Two DC sources : 1 kW each  
 One RF source : 600 W  
 Base pressure : ~ 10<sup>-8</sup> Torr  
 Target size : 75 mm dia

Substrate size : Max 75 mm dia  
 Substrate target distance : 4 -10 cm  
 Substrate temperature : room temp -800 °C  
 Different gas inlet : Ar, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>.

The operation of the system is fully automated through a personal computer and all the deposition parameter are displayed on the screen of PC and can be changed during operation. Figure 1 shows typical screen display during running of the system:

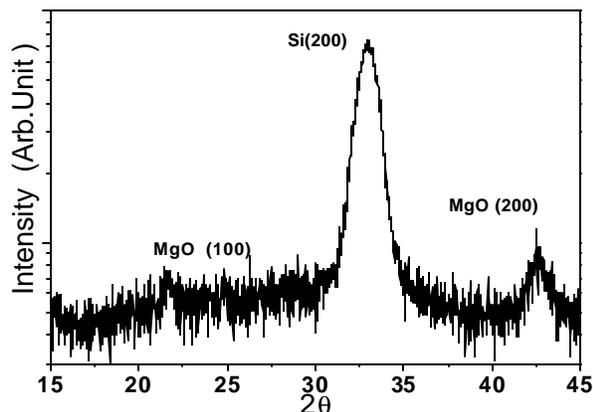


Fig.2 :X-ray diffraction of MgO thin film on single crystal Si(100) substrate deposited by RF magnetron sputtering.

To test the performance of the assembled magnetron sputtering system, a number of trial depositions of different materials such as Ti, Al, Si, W etc. were carried out in DC sputtering mode in vacuum as well as in reactive atmosphere. In order to test RF sputtering unit, deposition of MgO was carried out. Deposition was carried out on Si (100) substrate at 50 W RF power. MgO is an insulating system and normally one can get very low deposition rate (~ 0.1<sup>2</sup>/sec). We got better deposition rate compared to earlier reports. Figure 2 shows the X-ray diffraction pattern of deposited film. It can be seen that deposited film is of preferred orientation in (100) direction.

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## FACULTY SEMINARS AT CSR INDORE CENTRE

Speaker	Affiliation	Title	Date
Prof. B. S. Madhava Rao	Raja Ramanna Fellow, Department of Chemistry, University of Pune	Pulse Radiolysis: An Excellent Tool for Free Radical Research	30 Oct 2006
Dr. K. Sangunni	Associate Professor Physics Dept., IISc, Bangalore	Conduction Change in Chalcogenide Glasses	13 Sept 2006
Prof. Ullrich Pietsch	Physics Deptt, Universität Siegen, Germany	Molecular Magnetism in Supramolecular Structures	26 July 2006
Dr. Ravi Kumar	Materials Science Division, Inter University Accelerator Centre, New Delhi	Novel single-phase formation of Co-implanted ZnO thin films by swift heavy ion irradiation: Optical Studies	19 July 2006
Prof. Mrs. S.K. Kulkarni	DST Unit on Nano Science & Technology, Department of Physics, University of Pune	Size and Shape Controlled Small Particles	22 May 2006

## *New developments in research facilities at UGC-DAE CSR Kolkata centre* **Installation of a 1.5T magnet for transverse Mössbauer measurements**

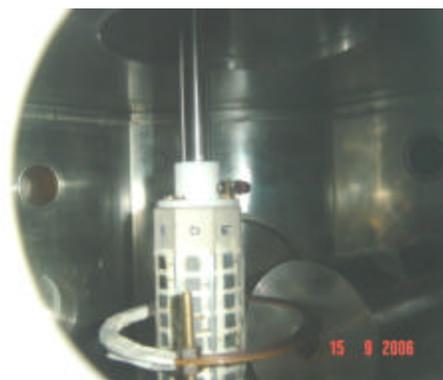
A 1.5T magnet has been installed and coupled to one of the Mössbauer spectrometers of the centre as shown in the figure below. This will enable one to record spectrum while the sample is in a transverse magnetic field at room temperature. Surface disorder and spin canting in some of the nano-composite samples prepared in the laboratory will be studied shortly using this facility. This facility is first of its kind in Eastern India and expected to be used by a large number of researchers from various universities and academic institutions. A cryostat will shortly be added to the facility and a 50 mCi Mossbauer source will be procured to facilitate measurements down to 20K.



## **TRACE ELEMENT STUDIES USING MULTI-TARGET OCTAGONAL LADDER**

Kolkata Centre has an extensive trace element research program in cross-disciplinary sciences involving physicists, chemists, biologists and environmentalists. Research and developmental efforts for designing of the experimental protocols for various inter-disciplinary areas for sample collection/ preparation and standardization have been carried out resulting in the formation of a cohesive program for trace element based multidisciplinary research. Trace element determination is carried out using the 3 MV Tandem accelerator at Institute of Physics, Bhubaneshwar and also using the EDXRF and AAS set-ups at the Kolkata centre. This multi-disciplinary research facility is one of the very few available to the university users in the country.

A major problem faced during the accelerator experiments was shortage of the available beam time demanding a better utilization of the expensive and hard-to-get accelerator time. This problem is specially severe for the cross-disciplinary studies as these studies require a large number of samples to be studied (~ 100 for each one such experiment). This problem has been solved by fabrication of a specially designed target ladders at the centre. The ladder with octagonal shape and off-axis mounting can load a total of 56 targets in the existing small vacuum chamber in one go. An additional identical ladder is kept ready with another set of 56 targets. In this way the effective beam time utilization has improved by almost an order of magnitude. Therefore, now in each beam time of 3 days, we are able to bombard ~ 300 targets. Figure below shows the target ladder outside and inside the scattering chamber during a recent beam time. With the precise design and fabrication, the target-detector geometry has also improved considerably.



A number of environment related studies have been carried out: metal distribution and accumulation in the wetland ecosystem in Bengal, a RAMSAR site and the role of biotic and abiotic components of the environment in bio-remediation of heavy metal burden; elemental profile of lichens as pollution indicator; water pollution studies in the Nainital Lake, characterization of fly ash in and around thermal power plants etc. Biological studies include study of trace elements in chemically induced hepatocarcinogenesis with emphasis on iron overloading; potential of herbal plants as anti cancer agent; effects of Se and Zn in As toxicity in male Wistar rats etc.

# Magneto optical Kerr effect for study of thin film magnetism

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## 1. Introduction

Magnetic thin films have been a topic of great current interest because of their novel physical properties and diverse technological applications. In investigating magnetic thin films, the magneto-optical Kerr effect (MOKE) has been widely utilized as a tool in probing surface magnetism. The surface Magneto-optic Kerr effect (SMOKE) made its debut as an experimental technique to study surface magnetism around 1985. The principle of the Kerr effect is defined as “when a plane polarized light interacts with a magnetic material the polarization gets changed by an amount proportional to the magnetization of the specimen” [1]. The effect in the reflection is known as Kerr effect and in the transmission it is known as Faraday effect. The MOKE, fundamentally related to the spin-polarized electronic band structure, manifests itself by the change of polarization and/or intensity of incident polarized light when it is reflected from the surface of a magnetized medium. The magneto-optical effects are shown to be proportional to the magnetization (M) in ferromagnets or a linear summation of sublattice magnetizations in ferrimagnets [2]. Using the

## 2. Experimental Details

Typical set-up of the MOKE system is shown in figure-1. In a simple configuration two polarizers are used as polarizer and analyzer. The polarized light from the first polarizer falls on the sample placed in a magnetic field. Because of the Kerr effect, the polarization of the reflected light will be different (proportional to magnetization) and this change in polarization can be measured using a second polarizer kept at cross to the first polarizer. The discussed crossed analyzer and polarizer method is prone to lot of noise due to stray light, vibrations and instability in the intensity of the laser. These background causes distortion to the hysteresis loop. A.C. modulation techniques in conjunction with the lock-in detection help in improving the S/N ratio. Several different devices have been proposed to modulate the state of polarization of the incident light such as, spinning analyzer prism and vibrating half shade polarizer which comes under mechanical modulators; Faraday modulators, Pockels cell and Photo-elastic modulators (PEM) which comes under electronic modulators. We have used PEM as a modulator. The advantages of this type of modulators are a large clear aperture, a short light path and a large radiation throughput. In addition the modulator can be used over a wide range of wavelengths. The details of the MOKE system at CSR are the following. Two Glan-Taylor prisms (with antireflection coating and extinction ration better than  $10^{-5}$ ) are used as polarizer and analyzer. Intensity stabilized He-Ne laser

Kerr effect one, therefore, probes a quantity proportional to the magnetization in a surface layer, the thickness of which is determined by the optical absorption coefficient of the material at the wavelength used for the experiments. For metals in the visible region this surface layer thickness is typically 10-20 nm. Magneto-optics is presently described in the context of either microscopic quantum theory or macroscopic dielectric theory [2,3]. Macroscopically, magneto-optic effects arise from the anti-symmetric, off-diagonal elements in the dielectric tensor. Microscopically, the coupling between the electrical field of light and the electron spin within a magnetic medium occurs through the spin-orbit interaction.

The present write-up does not deal with the theoretical aspects of MOKE, but rather presents experimental details, and some results with thin Fe film carried out in the longitudinal geometry, the advantages and disadvantages of MOKE and brief account of the different problems studied using MOKE.

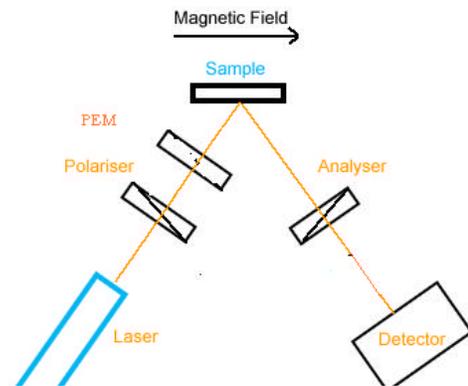


Figure-1. Block diagram of the MOKE setup

(632.8 nm), Si photodiode (active area  $\sim 25\text{mm}^2$ ) as detector and Stanford make (model SR-830) Lock-in amplifier are used for the set-up. PEM operating at a frequency of 50 kHz is used for the modulation. The locally fabricated electro-magnet can give up to 1.8 kOe in both longitudinal and polar geometries. For a p-polarized light, if the sample is non-magnetic, then the reflected light is purely ppolarized. However, if the sample is magnetic, then the reflected light consists of an s-component ( $E_s$ ) in addition to the dominant p-component ( $E_p$ ). Therefore, measuring this s-component

will be the goal of the experimental set-up. This is realized by keeping polarizer in front of the photo-detector to eliminate the pcomponent. Generally the second polarizer is kept slightly away from the extinction

( $\delta$ ). It can be easily shown that the intensity measured by the detector is,

$$I = \left| E_p \sin \delta + E_s \cos \delta \right|^2 = \left| E_p \right|^2 \left| \delta + E_s / E_p \right|^2 \\ = \left| E_p \right|^2 \left| \delta + \epsilon' + i\epsilon'' \right|^2$$

since both  $\delta$  and  $\epsilon''$  (Kerr rotation and ellipticity respectively) are linearly proportional to the magnetization, the measured intensity as a function of applied field (H) yields the magnetic hysteresis curve.

The direction of the magnetization vector with respect to the reflection surface and the plane of incidence categorize MOKE. MOKE can be done in three configurations viz., longitudinal, polar and transverse as shown in the figure-2. In the longitudinal geometry, the applied magnetic field will be parallel to the film surface and in the plane of incidence. In this geometry, the signal is sensitive to the in-plane component of the magnetization. In the polar geometry, the applied field will be perpendicular to the film surface and in the plane

of incidence. The signal is sensitive to the out-of-plane component. In the transverse geometry the applied field will be perpendicular to the film surface and also to the plane of incidence. In the first two cases, there will be a change in the state of polarization due to magneto-optical effects and in the transverse mode it is the intensity, which changes in proportional to the magneto-optical interactions.

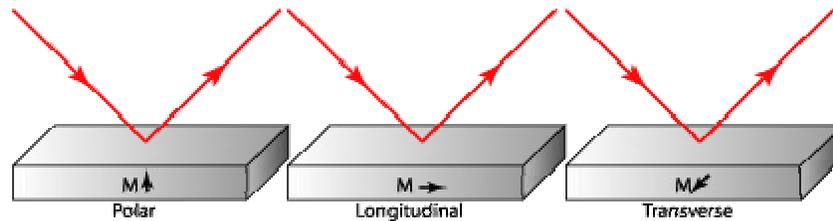


Figure-2. Various MOKE geometries

### 3. Results and Discussion

The important parameters of the system are angle away from extinction ( $\delta$ ), retardation of the modulator, angle of measurement etc. Out of the above three parameters,  $\delta$  is critical because when the second polarizer kept at any angle away from extinction can give a hysteresis loop which may not be a true characteristic of the sample. To determine the proper  $\delta$  value the following exercise has been done. The Kerr sensitivity defined as the difference in the intensity for a sample magnetized to saturation in opposite directions and normalized to maximum intensity is measured for a Fe film of structure Float Glass (substrate)/Fe(70 Å)/Au(20 Å) in the longitudinal mode and is shown in figure-3 (a). The maximum Kerr sensitivity occurs as the analyzer approaches  $90^\circ$ . It can be shown starting from the Malus law that the Kerr sensitivity is proportional to the tan of the analyzer angle. Even though the maximum sensitivity

is at extinction, in order to have detectable intensity it is necessary to use slightly away from extinction. The analyzer is kept  $\sim 2^\circ$  away from the extinction in all the measurements reported in the present paper.

Use of PEM enables to measure first and second harmonic components i.e., Kerr rotation and ellipticity simultaneously. The retardation of the PEM is dependent on the wavelength. Even though the used PEM is calibrated for the He-Ne at the factory, cross check of the actual and the set values of retardation is necessary periodically. The measured intensity at the detector output can be, using the Jones matrix of the optical elements involved, written as

$$I / I_0 = 1 + 4J_2(\mathbf{f})\mathbf{q}_k \sin 2\omega t - 4J_1(\mathbf{f})\mathbf{e}_k \sin \omega t + \dots$$

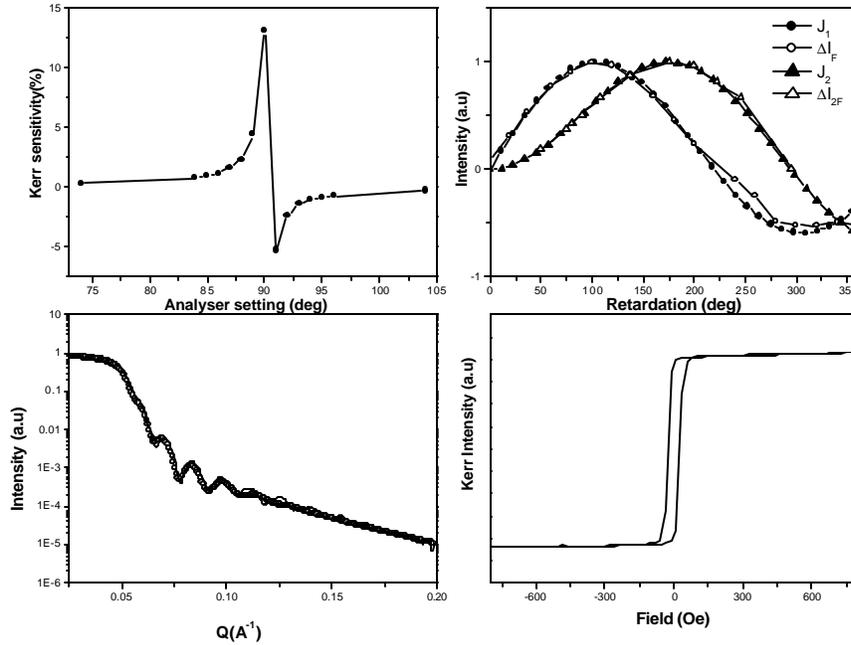


Figure-3. (a) The variation of Kerr sensitivity with the analyser setting (b) Variation of first and second harmonic components as a function of PEM retardation (c) X-ray reflectivity pattern of Si(substrate)/Fe(30nm)/C(2nm). The data is fitted (solid line) to obtain the exact thickness of the layer (d) MOKE hysteresis loop of Si(sub)/Fe(2.5nm)/C(2nm)

where  $\omega$  is the modulation frequency (50 kHz) and  $\phi$  is the retardation,  $J_1$  and  $J_2$  are the Bessel function of first and second order.

$\Delta I_F$  and  $\Delta I_{2F}$  were measured for a Float Glass (substrate) /Fe(100Å<sup>0</sup>) /Au(20Å<sup>0</sup>) film as the difference in the intensity of the first and second harmonic components with the application of magnetic field sufficient to saturate the sample in opposite directions for different retardations. The result is plotted in figure-3(b). The overlap between the measured and theoretical curves (i.e., the first and second order Bessel functions) clearly indicates no appreciable mismatch between the set and actual retardation. Further it can also be seen that one can set the retardation at a particular value for maximizing a particular harmonic component.

It is known that the magnetic properties of thin films depends on various parameters such as thickness of film, roughness, preparation conditions etc [4]. The aim of the present study is to show the sensitivity of the present MOKE system and also change in the signal depending on the thickness of the film. For this purpose Fe films of different thickness values viz., 2.5, 5, 7, 10 and 30nm on float glass and Si substrates are deposited using electron beam evaporation method under high vacuum conditions [5]. A capping layer of 2nm either

gold or carbon are used for protecting the sample from the oxidation. X-ray reflectivity (XRR) is used for the measurement of the thickness of the films. The XRR pattern of the 30nm Fe film is shown in figure-3(c). The solid line represents the best fit to the data with the Parratt formalism [6]. The obtained thickness of the film is  $30.5 \pm 0.1$ nm against the designated 30nm. To demonstrate the sensitivity of the MOKE measurement, hysteresis loop of 2.5nm Fe film is measured and is plotted in figure-3(d). The clear square hysteresis loop of the Fe film shows that it is possible to detect the magnetic properties of thin films down to very small thickness. It may be noted that the active ferromagnetic film in this may be less than that of the 2.5nm as it is expected that a ferromagnetic dead layer formation at the interface of the Fe film and the non-magnetic capping layer. The nearly square loop in the longitudinal mode indicates that magnetization is in the plane of the film. Further, to make sure that what is measured is not an experimental artifact, hysteresis loop for a non-magnetic film (silver) is measured and no loop is observed. Figure-4 shows the MOKE loops in the longitudinal mode for the Fe film of different thickness values taken with the same settings.

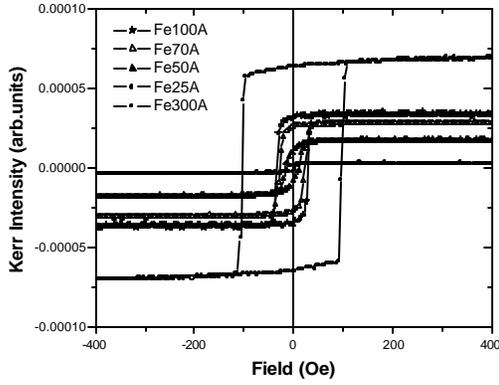


Figure-4. MOKE hysteresis loops of Fe films of various thickness values

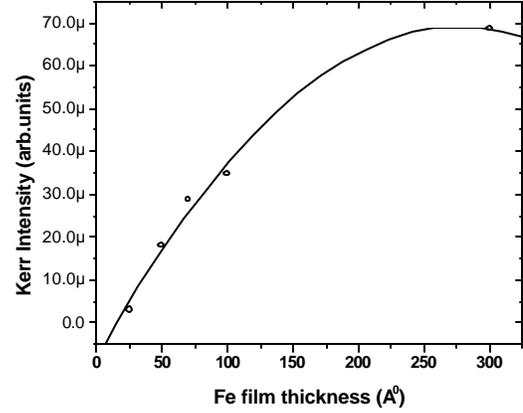


Figure-5. Variation of Kerr intensity as a function of Fe film thickness

Since the measurements are carried out with the same settings, it is possible to compare the intensity of the Kerr signal, which is proportional to the magnetization of the medium. The Kerr intensity shown in figure-5 is plotted as a function of the thickness of the film.

The initial linear increase in the intensity, as a function of thickness can be understood as following. The equation for the Kerr rotation in the longitudinal

geometry is given by [7] 
$$f_{long} = \left(4 \frac{P}{I} \frac{n_{sub}}{1 - n_{sub}^2}\right) Q d \sin^2 \theta$$
,

where Q is the magneto-optical constant, d is the thickness of magnetic layer and  $\theta$  is the angle of incidence measured from the surface normal. The above equation states that for a FM film deposited on the same substrate, the Kerr intensity is proportional to the thickness of the film and also to the angle of incidence. Saturation effects are observed for the Fe film of 30nm thickness. It is also observed that as the angle of incidence is increased, the signal intensity also increases. All the above measurements are carried out at a particular angle of incidence.

The present MOKE system is used extensively for the study of thin film magnetism. The following are few representative examples of such measurements. The formation of ordered  $L1_0$  FePt phase with the thermal annealing of Fe/Pt multilayers is evidenced from the increase in the coercivity values obtained from

longitudinal MOKE measurements [8] and the effects of ion beam irradiation in FePt system are also studied using MOKE [9]. The study of antiferromagnetic coupling in systems exhibiting GMR such as Fe/Cr /Fe trilayers is carried out [10]. Study of in-plane anisotropy [11] and the tailoring of magnetic anisotropy with the ion beam irradiation [12] is done. MOKE is used to study the magnetic changes with thermal annealing in Fe/Al [13,14], Ti/Ni multilayers [15], thin Co films on Si and GaAs substrates [16].

The surface magneto optical Kerr effect has got several advantages for the study of thin film magnetism as compared to SQUID, VSM such as the ease of measurement, sensitivity of the measurement etc. The only disadvantage perhaps is what we get from the measurement is not the absolute magnetic moment, but rather a quantity proportional to magnetization. However, calibrating the system can circumvent this problem.

In summary, experimental details, basic principle and result with thin Fe film are discussed in the present paper. Studies such as in-plane anisotropy, study of the wedge thin films with the controlled translation of the sample in the magnetic field are possible with the existing facility. Further efforts of using the MOKE system with low temperature, high magnetic field and during deposition of the film (in-situ) are in the process of development.

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## Pulsed laser deposition facility at UGC-DAE CSR, Indore

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Since for any technological application, integration of material into the thin film is the first step towards its realization. The contemporary technologies highly rely on the compactness; therefore, thin films turn out to be an imperative requirement for integrating various oxide layers. Moreover, thin films tender immense importance in study of their properties primarily because compared to the synthesization of single crystal bulk, thin films can be easily produced in single crystal form.

Various deposition techniques are known to synthesize thin films such as pulsed laser deposition (PLD), chemical vapor deposition (CVD), metal organic chemical vapor deposition (MOCVD), DC and RF sputtering, molecular beam epitaxy (MBE), thermal evaporation, sole gel etc. However, the basic process involved almost in all the techniques is to evaporate the material to provide atomic or molecular species in sufficient background pressure and carry them to the properly heated substrate of choice. The choice of substrate for the thin film deposition is guided by the lattice parameter of the compound and substrate as well as the orientation of the substrate. Among the various techniques mentioned above for thin film deposition, PLD is one of the most widely used thin film growth techniques. It has been established that high quality epitaxial films of several materials, including multicomponent oxides such as  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO) superconductors,  $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$  manganites, and several other materials can be grown using PLD.

The success of PLD technique lies in the fact that it preserves the target stoichiometry in the films, which has been found difficult for other conventional techniques such as evaporation, sputtering, etc. Another advantage of PLD technique over other techniques is that the operating pressure in the system is almost an independent variable and does not affect the evaporation process significantly since the laser source is independent of the deposition chamber. Excimer lasers such as XeF ( $\lambda = 352$  nm); XeCl ( $\lambda = 308$  nm); KrF ( $\lambda = 248$  nm); KrCl ( $\lambda = 222$  nm); ArF ( $\lambda = 193$  nm); and  $\text{F}_2$  ( $\lambda = 157$  nm) are commercially available and can be used for thin film deposition. It uses a pulsed laser beam, usually but not necessarily, from an ultraviolet excimer laser with pulse energy of about 1 J. The typical duration of the laser pulse is a few tens of nanosecond. Because of such a short duration of pulse, tremendous power ( $\sim 10 - 100$  MW/pulse) is delivered to the target. This is because of the nonequilibrium nature of PLD in the sense that the absorption of energy and ablation takes place in a very short time, usually within a nanosecond, before thermodynamic equilibrium is reached. We have KrF excimer laser in our UGC-DAE CSR, Indore center (Fig. 1), which is fully functional and being extensively used by internal as well as university users.

Typical schematic of the pulsed laser deposition technique is shown in Fig. 2. The laser beam of desired energy density is made to incident on the well sintered palletized target material (approximate diameter 1") at an angle of  $45^\circ$  with the target surface. The target is rotated with the help of a stepper motor in order to avoid crater formation on the target due to ablation. The desired energy density is obtained by focusing the laser beam with the help of quartz lens to a proper spot size. Generally for most of the oxide systems (manganite, cuperate, ferrite, magnetite etc) laser energy density is maintained at  $\sim 2$  J/cm<sup>2</sup>. The substrate is mounted in front and parallel to the target at a distance of 4-5 cm on a resistive heater with the help of thin conducting silver layer or clamp. The temperature of the substrate can be measured using a calibrated thermocouple placed on the substrate or on separate small piece of substrate itself. Before mounting the substrate, it is properly cleaned by standard route in the sequence of trichloro ethylene, acetone and methanol in ultrasonic, each for five minutes.

When a laser beam of energy density above a material dependent critical value is incident on the target, a large amount of energy is deposited in a few hundred-nanometer depth from the surface over a very short time scale. Due to such a transient energy transfer, temperature of the surface layers is raised to a sufficiently high value (higher than the melting temperature of the material) and thus the melting of material starts at the surface. The process takes place in a very short duration of time scale. This causes a rapid ejection of the laser-induced plasma of materials at right angle from the surface of the target in the forward direction towards the substrate. The plasma plume contains various excited atoms, molecules, ions and neutral species. This plasma quickly expands away from the target towards the substrate where the adiabatic expansion of

plasma at the surface of the heated substrate takes place leading to growth of the desired thin film. A view of the plasma expansion towards heating substrate after the laser target interaction is shown in Fig. 3.



Fig. 1: Photograph of pulsed laser deposition system at UGC-DAE CSR, Indore.

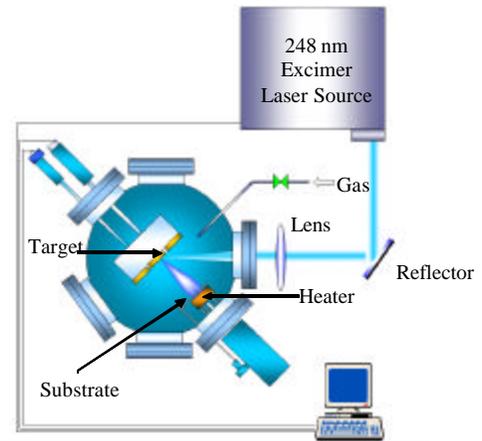


Fig. 2: A schematic view of pulsed laser deposition technique.

PLD is one of the most successful growth techniques for the deposition of complex or multicomponent materials. However, it suffers from certain drawbacks such as difficulty in large area film deposition due to narrow angular distribution of the plasma plume. To overcome this problem, substrate can be scanned vertically/horizontally or laser beam can be scanned over the large area of the target. Another major disadvantage is the “splashing effect” causing large particulates to nucleate on the film. This causes significant surface roughness. Sanding the target before deposition and continuous scanning of the target during deposition can minimize this effect.

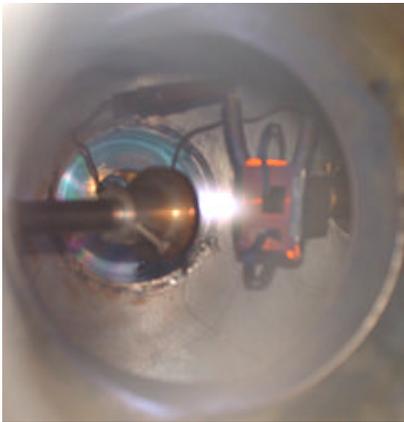


Fig.3: A view of plasma formed during pulsed laser deposition of Fe<sub>3</sub>O<sub>4</sub> thin film

While PLD is recognized to transport the stoichiometry from the target to the film, there are several parameters, which immensely influence the growth and properties of thin films. These parameters are laser energy density falling on the surface of the target, ambient background pressure during and after deposition, pulse repetition rate, temperature of the substrate, choice and orientation of the substrate itself, target to substrate distance etc. Laser energy density is a vital factor, which hugely affects the properties of films. If the energy density is low, the complex target molecules do not evaporate congruently; if very high, droplet kind of particles is deposited on the substrate. The ambient oxygen partial pressure too is vital for the thin film growth of oxides to make up the loss of oxygen in the ceramic target itself or during the course of transfer of the excited species from the target to the substrate. Here we present some of the results on Fe<sub>3</sub>O<sub>4</sub> thin films on various substrates deposited using our PLD set up. We have deposited thin films of Fe<sub>3</sub>O<sub>4</sub> on (100) oriented MgO substrate by pulsed laser deposition technique.

Films are epitaxial and highly oriented along c-axis {Fig. 4 (a)} and show characteristic Verwey transition in resistivity and magnetization data {Fig. 4 (b)}.

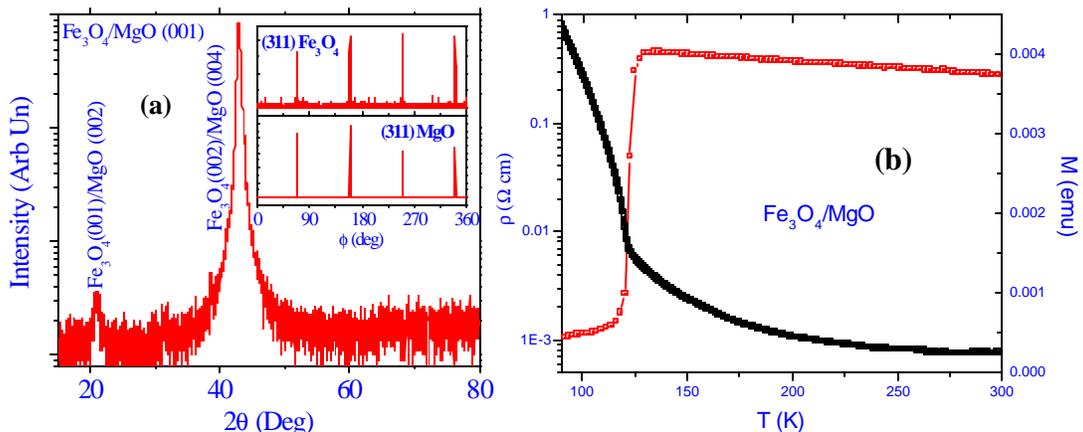
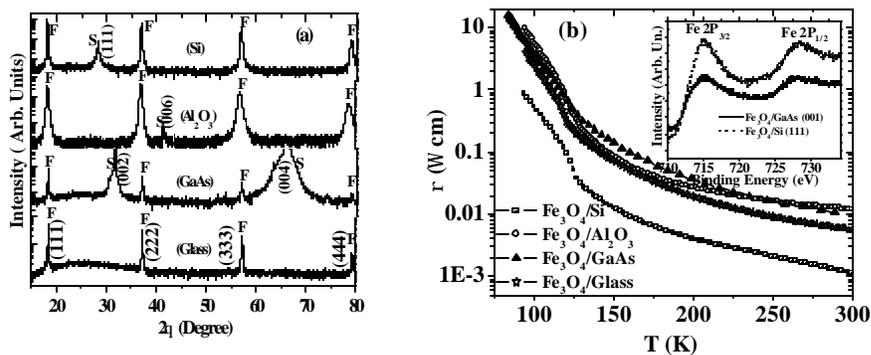


Fig. 4: (a) XRD pattern of pulsed laser deposited Fe<sub>3</sub>O<sub>4</sub> film on single crystal MgO (100) substrate, inset shows the in-plane epitaxy of the film and (b) resistivity and magnetization behavior as a function of temperature.

Compatibility between  $\text{Fe}_3\text{O}_4$  and technological important semiconducting substrates like Si or GaAs substrate is also explored. As shown in Fig. 5 (a) the preferential oriented growth of  $\text{Fe}_3\text{O}_4$  film is along (111) direction on various substrates. These results indicate that the easy axis growth direction of  $\text{Fe}_3\text{O}_4$  is (111). Indeed, this is further confirmed by preferential (111) oriented growth of  $\text{Fe}_3\text{O}_4$  film on amorphous substrate like float glass. They also reveal the characteristic Verwey transition in their resistivity versus temperature behavior {Fig. 5 (b)}. X-ray photo spectroscopy (XPS) spectra of these films are similar to that for  $\text{Fe}_3\text{O}_4$  bulk single crystal, confirming the  $\text{Fe}_3\text{O}_4$  phase in the film {inset Fig. 5 (b)}. To the best of our knowledge this is the first report which establishes the compatibility of oriented crystalline  $\text{Fe}_3\text{O}_4$  thin films on various single crystal substrates including amorphous substrate without requiring any buffer layer.

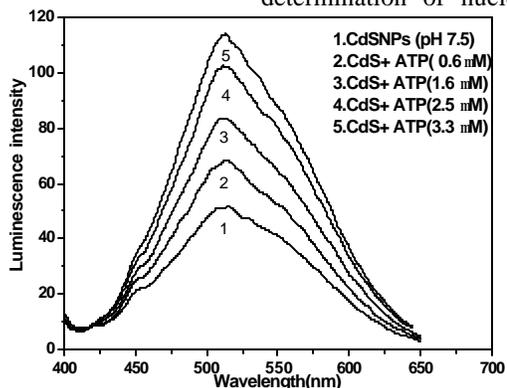


**Fig. 5:** (a) X-ray diffraction pattern for  $\text{Fe}_3\text{O}_4$  films on different substrates, film and substrate peaks are marked as F and S respectively; and (b) Resistivity behavior of these films, inset shows the XPS spectra for  $\text{Fe}_3\text{O}_4$  films on Si (111) and GaAs (001) substrates.

## LUMINESCENCE ENHANCEMENT OF BIOFUNCTIONALIZED CDS NANOPARTICLES BY NUCLEOTIDES : A STEP TOWARDS DEVELOPMENT OF ATP SENSOR IN BIOLOGICAL SYSTEMS

Bioconjugation of nanoparticles, i.e., their attachment to biospecific ligands yields unique hybrid materials. They incorporate optical/electrical properties of nanoparticles and display highly selective binding to oligonucleotides and proteins. A study of attaching fluorescent CdS nanoparticles to the biological macromolecule DNA has been carried out at the Kolkata centre to explore possibilities of developing fluorescent nanoparticle-probe for quantitative estimation of the DNA bases.

Highly luminescent CdS nanoparticles (avg. diameter 3.8 nm) were synthesized and were biofunctionalised with cysteine. There were used for the interaction study with DNA bases and corresponding nucleotides. It was found that the enhancement of luminescence at pH 7.5 and 10.5 is specific for adenine. Other nucleobases do not cause any perceptible change in the photoluminescence of the nanoparticles either at basic or neutral pH. (pls. see the figure). The linear portion of the relative fluorescence enhancement ( $I_0/I$ ) plot with increasing concentration of nucleotides could be utilized for the quantitative determination of nucleotides in biological samples. Binding constants of different nucleotide and adenine, calculated using Langmuir adsorption isotherm clearly indicate that adenine and ATP bind more strongly to the surface of the CdS nanoparticles as compared to other nucleotides. FTIR and Raman spectroscopic data confirm the specific binding of adenine and ATP at the nanoparticle surface.



The above demonstrated interaction specificity of adenine with CdS nanoparticles could be immensely helpful in developing new protocols for fluorescence based biosensors in near future. In certain pathological conditions such as adenosine deaminase (ADA) deficiency and adenine phosphoribosyl transferase (APRT) deficiency, there is abnormal rise in concentration of adenine in body fluids and that of ATP in erythrocytes of patients which leads to chronic renal failure. The enhanced sensitivity of luminescence of CdS NPs for ATP in micromolar range permits determination of the ATP-concentration in the diseased erythrocyte cells. Valuable information emanating from these studies can also help us design fluorescent nanoparticle probes for detection of nucleic acids having higher adenine content.

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