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## ECR Based Low Energy Ion Beam Facility at VECC, Kolkata

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**Abstract.** A low energy heavy ion irradiation/implantation facility has been developed at VECC, Kolkata for materials science and atomic physics research, utilizing indigenously developed 6.4 GHz ECR ion source. The facility provides high charge state ion beams of N, O, Ne, Ar, S, Kr, Xe, Fe, Ti, Hf etc. up to a few micro amperes to an energy of 10 keV per charge state. The beam energy can be further enhanced by floating the target at a negative potential (up to 25 kV). The ion beam is focused to a spot of about 2 mm diameter on the target using a set of glaser lenses. A x-y scanner is used to scan the beam over a target area of 10 mm x 10 mm to obtain uniform implantation. The recently commissioned multi facility sample chamber has provision for mounting multiple samples on indigenously developed disposable beam viewers for insitu beam viewing during implantation. The ionization chamber of ECR source is mainly pumped by ECR plasma. An additional pumping speed has been provided through extraction hole and pumping slots to obtain low base pressure. In the ion source, base pressure of  $1 \times 10^{-7}$  Torr in injector stage and  $\sim 5 \times 10^{-8}$  Torr in extraction chamber have been routinely obtained. The ultra-high vacuum multi facility experimental chamber is generally kept at  $\sim 1 \times 10^{-7}$  Torr during implantation on the targets. This facility is a unique tool for studying fundamental and technologically important problems of materials science and atomic physics research. High ion flux available from this machine is suitable for generating high defect densities i.e. high value of displacement-per-atom (dpa). Recently this facility has been used for studies like "Tunability of dielectric constant of conducting polymer Polyaniline (PANI) by low energy Ar<sup>9+</sup> irradiation" and "Fe<sup>10+</sup> implantation in ZnO for synthesis of dilute magnetic semiconductor".

### 1. Introduction

In the present day, immense importance has been concentrated into the Materials Science Research with thin films, regarding basic physics as well as applications point of view. Exotic physics comes out from defects and disorders in two dimensional systems. Hence, charged particle irradiation brings forth enormous novel phenomena in thin films. Low energy (hundreds of keV) heavy ions from ECR ion source are ideal tools for generation of tailor made defects in thin films. The low energy nuclear projectile experiences significant energy loss through collisions in the target and its range may vary up to a few hundred Nano-meters. It causes an enormous generation of point defects through the so called nuclear energy loss in addition to the significant inelastic energy loss. Low energy ion beams also

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provide a controlled means for modifying material surfaces for achieving desired properties. Recently, a low energy heavy ion irradiation/implantation facility has been developed at VECC, Kolkata for materials science and atomic physics research.

The facility comprises of a 6.4 GHz electron cyclotron resonance (ECR) ion source [1] which is a special types of hot plasma machine to produce multiply charged ions, a magnetic charge-state analyser, focussing magnets in the beam transport line and a multi facility implantation chamber. The total vacuum space of the facility is approximately 176 litres which is spread over a large length and area. Total length of the vacuum system is segmented and conductance limited by slits, collimators and diaphragms. The whole volume is pumped by several distributed pumping stations isolated by ultra-high vacuum gate valves. Besides magnetic confinement, operating frequency and microwave power of the ion source, the vacuum plays a major role in multiply charged ion beam production, analysis and transport. In ECRIS plasma discharge takes place at a vacuum level of  $\sim 10^{-7}$  Torr. The low-pressure condition is essentially needed to minimize charge exchange loss due to recombination of multiply charged ions with the lower charge state ions and the neutral atoms. A fixed ratio of neutral to electron density must be maintained for optimum production of a particular charge state in the steady state condition. As the electron density is proportional to square of the injected microwave frequency ( $n_e \propto f^2$ ), it is understood that a particular operating pressure is essential for a specific charge state generation. The requirement of good vacuum in the beam extraction, analysis, transport and implantation area is again essential to minimize the space charge effect, beam scattering and implanted impurity caused due to residual environment.

## 2. The implantation facility

A low energy heavy ion implantation facility as shown in figure 1. has been developed at VECC using the indigenously built 6.4 GHz ECR ion source [VECECR IS] [1-3]. The ion source was basically designed to meet the injection parameters of the cyclotron and it operates with the maximum extraction potential of 10 kV, thus delivering ion beams at a maximum energy of 10 keV per charge state. The ion beam extracted from the ECR ion source is  $q/m$  analysed by a  $90^\circ$  double focussing analysing magnet with a resolution of 1 in 40 for 10 mm slit width. The analysed beam is optimized on a retractable type Faraday Cup through  $10 \times 10$  mm slit. The multiply charged ion beam is then allowed to propagate through an ultra high vacuum beam line up to the multi facility experimental chamber for carrying out implantation and other experiments.



Fig. 1. Layout of ECR-based Low Energy Ion Beam Facility

### 2.1 VECECR Ion Source

VEC-ECRIS is a single stage high magnetic field (High-B) ion source with a biased electron repeller placed on the axis, near the injection mirror point [2]. The supply of cold electrons and use of low mass mixing gas improve the source performance and stability. High-B VEC-ECR has only two sets

of coils with individual iron return yoke [Figure 2]. This topology facilitates with an easy tuning of the source and has negligible influence of the injection field on the extraction area due to an excellent isolation between the mirrors.  $B_{min}$  field is reduced but the peak fields are increased to 7.2 and 5.5 KGauss corresponding to mirror ratios to  $R_1=6.4$  and  $R_2=5$  respectively.

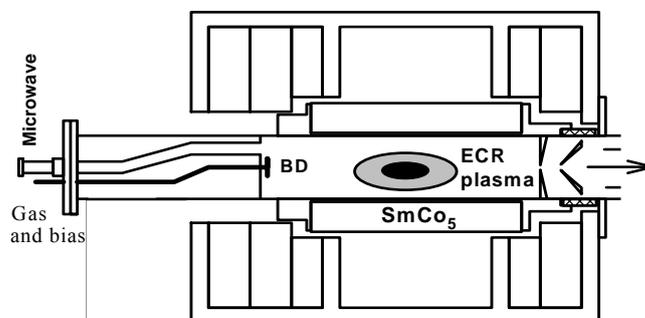
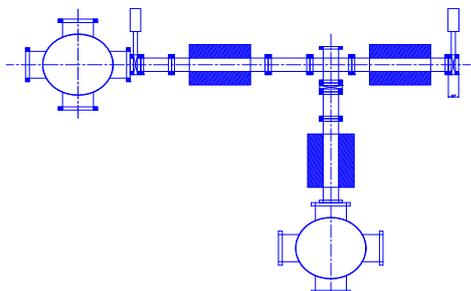


Fig. 2. ECR ion source schematic

### 2.2 Beam Transport and Experimental Facility

The mass analyzed ion beam is focussed by a pair of magnetic glaser lenses on the target film. The schematic of the beam line is shown in figure 3. Figure 4. shows the beam optical simulation result. The photograph shown in figure 5 is the beam spot seen on a beam viewer. The observed beam spot size (2mm dia.) shows a good co-relation with the simulation value.

#### Material Science



#### Atomic Physics

Fig. 3. Test chamber and beam lines

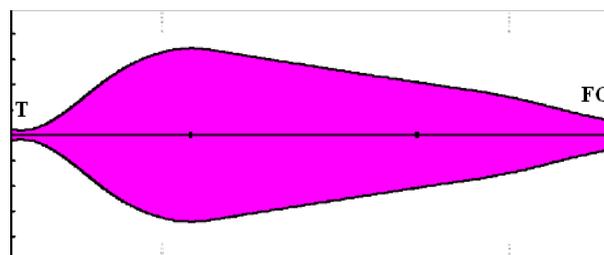


Fig. 4. Beam profile from faraday cup to target



Fig. 6. Multifacility experimental set up



Fig. 5. Beam spot on a viewer at target ladder

The details of the experimental setup are shown in figure 6. At the top flange of the multi-facility experimental chamber, a four axes sample manipulator has been commissioned to accurately positioning an electrically isolated target ladder assembly for mounting multiple targets at a time. Each sample sits over a disposable type indigenously developed low energy beam viewer for viewing the beam during implantation.

With a view to deliver enhanced variable ion energy, the provision has also been made to float the target up to a variable potential of  $-25\text{kVolts}$ . For most of the ground state experiments, total fluence is usually measured directly on a current integrator. But, in case of floating samples, to achieve higher projectile energy, the beam current is measured by an indirect method. Here also the total fluence is measured on the current integrator by monitoring the ion current captured by the collimator placed before the electrostatic scanner. For each such experiment, the collimator current, after scaling to a pre-calibrated factor, has been utilized as the total fluence measurement. During implantation on the metallic targets, the secondary electron produced from the target causes an appreciable error to the implantation doses. A suitable non-magnetic, 180 mm diameter semi cylindrical electron capture electrode has been kept isolated at 85 mm away from the target ladder assembly (figure 7a, b, c) to collect the produced secondary electrons. The secondary electron current thus produced is added algebraically to measured target current for actual fluence measurement. This arrangement facilitates us to measure the energy of the secondary electron in bulk or in discrete levels as required. This semi cylindrical electrode has two holes at a separation of  $45^\circ$  on the horizontal beam propagation plane. The 20 mm diameter hole which is concentric to the beam propagation centre allows the scanned beam for implantation on the target and another hole of 30 mm diameter enables us to online viewing the beam with the help of a CCD camera.



Fig. 7a. Target ladder assembly



Fig. 7b, c Integrated assembly of Collimator, beam scanner & electron capture electrode ( $180^\circ$  views)



Since the irradiation/implantation is motivated for the modification of the properties of materials with proper understanding of the mechanism, it is essential to have uniform irradiation over the target area as well as the thickness. Uniform irradiation along the thickness is guaranteed by thin films. But to have an uniform irradiation over the whole area of the sample, it is necessary to scan the beam over the target surface, preferably with very low time periods compared to the irradiation time. Based on the beam transport calculations and also considering the ion optical properties of low energy beam to achieve small beam size on the target, the final focusing glaser solenoid has been placed nearest to the test chamber. An electrical scanner with a changeable collimator assembly has been designed and developed to scan the beam on the thin target films at higher scanning rate. The scanned area can be finely adjusted and shifted electronically in X-Y directions. To satisfy all the above mentioned requirements, an integrated collimator, electrical scanner assembly along with a well separated secondary electron capture electrode have been designed and developed in our laboratory (figure 7b, c). The integrated assembly is housed inside the test chamber concentrically to the floating target assembly. This set up has been used regularly for carrying out several experiments.

### 3. ECR source vacuum and charge state production

Ionization takes place when sufficiently energetic electrons collide with neutral molecules under certain environmental conditions. Single impact and successive ionization are the two basic ionization processes to be mentioned. But the probability of single impact ionization is small compared to that of successive ionization process regarding multiply charged ion production. In  $B_{\min}$  field trap, the plasma is stable and the diffusion time of the ions is high enough to ionize the atoms to high charge state by successive ionization process. This process needs some time which is almost equal to the confinement time ( $\tau_i$ ) or exposure time of the ions to a cloud of hot electrons of density  $n_e$  with an impact energy  $T_e$ . The equilibrium charge state distribution is obtained by the balance between ion production rate and loss rate. In the steady state, the rate of change of ion charge state  $i$  is zero [4].

$$\frac{dn_i}{dt} = 0 = n_e (\sigma_{i-1, v_e} v_e) n_{i-1} + n_0 (\sigma_{0, i+1} v_{i+1}) n_{i+1} - n_e (\sigma_{i, i+1} v_e) n_i - n_0 (\sigma_{0, i} v_i) n_i - \frac{n_i}{\tau_i} \quad (1)$$

where  $n_e, n_0, n_i$  are the density of electron, neutral particle and ion of charge state  $i$  respectively

$\tau_i$  = ion confinement time  $\cong$  ionization time  $\cong$  the life time of ion for ionization of  $i$  th state,

$v_e$  = electron velocity,

$v_i, v_{i+1}$  = ion velocities for charge state  $i$  and  $i+1$  respectively,

$\sigma$  = average ionization cross-section for respective cases ( $10^{-16}$  to  $10^{-20}$  cm<sup>2</sup>).

The probability of dropping charge state by electron pick up is almost negligible compared to the probability of the charge exchange loss of produced ions colliding with low charge state ions and neutrals.

In the charge exchange process an ion and a neutral atom collide to form a molecular ion which then splits into two ions.



The cross-sections ( $\sigma_x$ ) of the recombination process are very large and increase with  $z$ .

The charge exchange cross-section formula given Chibisov may be written as:

$$\sigma_x = 1.7 \times 10^{-15} z \text{ cm}^2 \quad (3)$$

As the charge transfer time limits the life time ( $\tau_i$ ) of a cold ion of charge state  $z$ , it may be written as:

$$\tau_i \approx \tau_{z \rightarrow z-1} \approx [n_0 v_i \sigma_x]^{-1} \approx 5 \times 10^8 \frac{\sqrt{A}}{z n_0} \quad (4)$$

where  $n_0$  = neutral density (cm<sup>-3</sup>),

$A$  = the atomic mass number

$$v_i \approx \frac{10^6}{\sqrt{A}} \text{ cm s}^{-1} \text{ assuming ions are at thermal energy } T_i \approx 1 \rightarrow 5 \text{ eV}$$

The well-known criterion for stepwise ionization may be written as:

$$\xi n_e \tau_i \geq 5 \times 10^4 (T_e^{opt})^{\frac{3}{2}} \quad (5)$$

where  $\xi = \sum_{j=1}^N q_j$  = the total number of electrons in the outer shell

$T_e^{opt} \approx 5W_j^{\max}$  = the optimum electron temperature

The optimum plasma electron temperature is taken as five times the threshold energy  $W_j^{\max}$ . It is easily understood from the above equation that totally stripped light ions need

$$n_e \tau_i \sim 1.76 \times 10^{10} \text{ cm}^{-3} \text{ s for electron temperature } T_e^{opt} \sim 5 \text{ keV}.$$

To attain a charge state  $z$  the charge transfer time must be greater than the ionization time for the same  $z$  for a specified electron density  $n_e$ . When this condition is imposed to the stepwise ionization

criterion, the upper limit of neutral density  $n_0$  in the plasma is obtained for a specified electron density as:

$$\frac{n_0}{n_e} \leq 10^4 \xi [T_e^{opt}]^{-3/2} A^{1/2} z^{-1} \quad (6)$$

From this equation one can calculate the neutral density and the operating pressure of the discharge chamber for any charge state  $z$  at a particular electron density. For the ionization of hydrogen like ions  $\xi = 1$ . To produce  $Ne^{10+}$  in 6.4 GHz VEC-ECR a maximum typical value of electron density ( $n_e$ ) can be taken as  $n_{ce} \approx 5 \times 10^{11} \text{ cm}^{-3}$  [5,6]

$$T_e^{opt} \approx 5W_j^{\max} \approx 5keV, \text{ hence } \frac{n_0}{n_e} \leq 10^{-2} \text{ and } n_0 \leq 5 \times 10^9 \text{ cm}^{-3}$$

This corresponds to a pressure of  $1.4 \times 10^{-7}$  Torr. For  $Ar^{18+}$  the needed vacuum is about  $5 \times 10^{-8}$  Torr. So a higher frequency source operating at a higher magnetic field should give the same charge state production performance at a comparatively higher pressure. In a study [7], the vacuum requirements of ECR sources operating at different frequencies is shown in Figure 8.

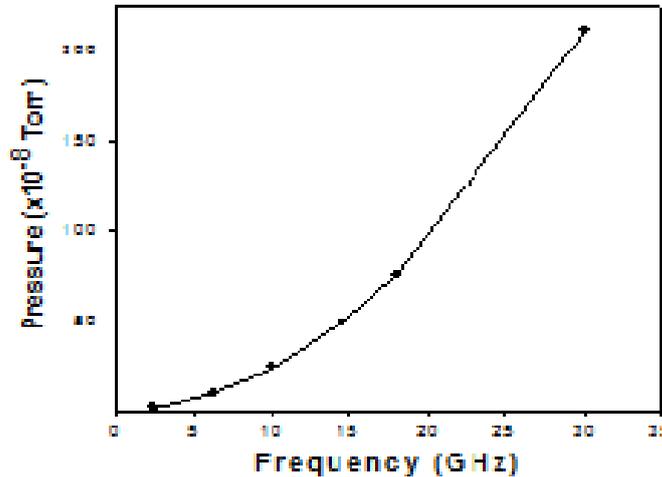


Fig. 8. Frequency vs operating pressure of ECR source [6]

Hence, the operating vacuum of the ionizer is extremely important parameter for high charge state production. The operating vacuum of the ionizer in the range of  $10^{-7}$  Torr is essentially needed to reduce charge exchange loss and the pressure in the ion extraction chamber should be maintained lower than  $10^{-7}$  Torr.

The main plasma cavity of the ECR ion source is 345 mm long and 108 mm in diameter which is made out of oxygen free high conductivity copper billet to meet the suitable surface condition essentially needed for good vacuum and also to satisfy the microwave properties. The chamber is water-cooled and carries 305 mm long rare earth sextupole magnet bars externally. The entire volume of the ion source is pumped by two 900 l/s special type oil diffusion pumps to achieve  $5 \times 10^{-8}$  Torr. Usually main plasma chamber is pumped by the plasma itself. Moreover an additional pumping speed (a few l/s) is provided through extraction hole and slanting pumping slots of the plasma orifice. The diffusion pumps are electrically isolated by ceramic tubes of low out-gassing rate and provided with a specially designed high voltage corona shield to avoid contamination by oil vapor fragmentation components. A pressure of about  $1 \times 10^{-7}$  Torr in the beam transport line is routinely obtained through distributed pumping by three turbo-molecular pumps to minimize scattering and charge exchange loss during beam transport.

#### 4. Experimental vacuum chamber

As the facility has been developed for carrying out research related to atomic physics and material science with low energy beam, the sample/target essentially should be thin films and the typical target modification depth is few tens to hundreds of nano-meters. Usually the scattering loss will occur. Depending upon the vacuum level there exists usual scattering loss to some extent. Beside this, there is the strong possibility that the chamber inner surface will be covered by the adsorbate which eventually will be buried into the film by the impact of the projectile ions. It introduces impurity in the implantation process producing erroneous result. The monolayer formation time basically can give us an idea about the desired vacuum. The monolayer time  $T = \frac{1.94 \times 10^{-6}}{p}$  sec. Where  $p$  is the

pressure in Torr. It is clearly understood that single monolayer is formed within two seconds time at a pressure of  $\sim 1 \times 10^{-6}$  Torr. The impurity mixing rate will be reduced to one tenth due to an improvement of the vacuum by an order of magnitude. Basically the test chamber should be a beakable ultra high vacuum chamber. The multi-facility chamber used in the present facility is a similar one, which can produce ultimate vacuum of  $3 \times 10^{-9}$  Torr by continuous pumping.

#### 5. Gas and Element Feeding System of ECR Source

Initially, the journey of the ion source was started to produce ions of the gaseous elements. However, due to the pressing demand of the materials scientists to use this facility, the ion beams of S, Fe, Ti and Hf have been developed using the MIVOC technique. In this method the source feed material is usually an organo-metallic volatile compound containing the element of the desired ions.



Fig. 9. MIVOC setup

We have observed that for obtaining good yield the vapour pressure of the compound should lie between 10 to 0.1 Torr at room temperature. A dedicated set up has been developed for this purpose which has the pumping and vacuum measuring facility of its own. This has been developed with a view to reduce the residual constituents and also to estimate approximately the vapour pressure of the compounds. The entire MIVOC setup is electrically isolated from the ion source, which enables its operation at ground potential. The vapour of the MIVOC compound is injected into the ion source using an electro-mechanically operated sapphire seal valve. With this arrangement it is possible to precisely control the elemental feed and one can optimize the ECR parameters for the desired ion species [figure 9]. We have observed that for certain ion species a suitable mixing gas is essential to

improve the required beam yield. The facility exists to inject simultaneously three sample elements which is needed for very special experimental purposes.

## 6. Recent Experiments

The ion currents for some species are given in Table 1 below. Using this facility, several ion implantation experiments have been carried out. Some of these experiments were aimed to simulate the modifications and damages created due to very high dpa in nuclear structural materials. Because of the availability of very high currents from this machine (~10 $\mu$ A) such experiments could be carried out successfully. In addition to this, implantation experiments on semiconductors, organic semiconductors, organic single crystals for non linear optics etc. have been carried out. To mention a few are given below:

1. Experiments were carried out for the synthesis of  $\beta$ -FeSi<sub>2</sub> by implantation of Fe-ions in single crystalline silicon samples.  $\beta$ -FeSi<sub>2</sub> is a direct band gap semiconductor having a large number of applications in optoelectronics[8].
2. In an experiment, the Fe<sup>10+</sup> ion were implanted in ZnO for synthesis of p-type ZnO [9].
3. Several charge states of Oxygen ion were implanted in PMMA samples to study the effect of charge states on the nature of material modification [10].
4. In another experiment, the tunability of dielectric constant of conducting polymer Polyaniline(PANI – Polyvinyl Alcohol (PVA) has been observed by low energy Ar<sup>9+</sup> irradiation. The modified material can be used as the e-m shielding materials [11].

Users from several universities [12,13], BIT Mesra [9], IIT Rorkee[10], UGC DAE CSR [8], BARC and VECC [11,14,15] have utilized this facility for carrying out several successful experiments

**Table 1.** Ion Current of different species

Species	Charge States	Typical Currents ( $\mu$ A)
Ne	6 <sup>+</sup> to 9 <sup>+</sup>	45 -1.5
Ar	8 <sup>+</sup> to 13 <sup>+</sup>	100-1.5
Kr	12 <sup>+</sup> to 19 <sup>+</sup>	45-6.5
Xe	20 <sup>+</sup> to 31 <sup>+</sup>	4 -1
C	2 <sup>+</sup> to 5 <sup>+</sup>	100-20
O	5 <sup>+</sup> to 7 <sup>+</sup>	120-12
N	4 <sup>+</sup> to 6 <sup>+</sup>	130-31
Fe	10 <sup>+</sup> & 11 <sup>+</sup>	10
Ti	11 <sup>+</sup>	7
S	10 <sup>+</sup>	2.5
Hf	12 <sup>+</sup> -29 <sup>+</sup>	5 -0.3

## 7. Conclusions

Low energy ion beam implantation has been proved to be an extremely valuable tool for atomic physics, surface physics and material science research. Ion loses energy by colliding with the atom of the material and displacing them from their lattice position generating enormous defects creating drastically new properties. Nuclear energy loss principally dominate at this energy regime. ECR based low energy facility can deliver very high ion currents for various elements at variable energies and very high value of DPA can be achieved easily for carrying out material damage experiments. Due to the low ion energy, the vacuum requirement for the whole facility is equally stringent as the production of multiply charged ions and delivering it on the target. A sufficiently adequate volume of

the multi-facility chamber prompts us to incorporate many more on line experimental facility in the set-up. Facility for on line IR temperature profiling and the heating/cooling of the samples at desired temperature will be commissioned very soon.

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