Epitaxial Crystallization

Ion implantation process provided the ways to tailor the conducting properties of semiconductors and thereby initiated the transistor revolution in electronics. During implantation of the desired element in a semiconductor, ion-induced collisions produce athermal atomic movements at and around the surface or interface, which can be controlled by varying the temperature and ion-beam characteristics, guiding the system between non-equilibrium and quasi-equilibrium states. Subject to the ion implantation process, high degree of damage formation due to collision cascades can lead to amorphization in semiconductors like Si and Ge. At temperatures where defects are mobile and interact, irradiation can lead to layer-by-layer amorphization, whereas at higher temperatures irradiation can lead to the recrystallization of previously amorphized layers.

Silicon-on-insulator (SOI) structure is used in the integrated circuits. SOI refers to the use of a layered silicon-insulator-silicon substrate in place of conventional silicon substrates in semiconductor manufacturing, especially microelectronics, to reduce parasitic device capacitance and thereby improving performance. SOI-based devices differ from conventional silicon-built devices in that the silicon junction is above an electrical insulator, typically silicon dioxide.

SOI structures are typically synthesized by implantation of oxygen ions in silicon (at elevated temperatures), which gives rise to amorphization, introduces roughness, and produces defects. Various annealing methods like furnace, laser and electron-beam have been employed to either recover the damage or to recrystallize the amorphous layers. In these processes annealing temperatures greater than $1000^\circ$C are generally needed. With the reduction in the size of the devices to submicron scales it has become a challenge to regain the lattice structure at lower temperatures to avoid undesired diffusion of the dopants [1]. It can be achieved in silicon at lower temperatures (only a few hundred $^\circ$C) by simultaneous irradiation with energetic ions [2]. This recrystallization process, achieved by ion-atom collision, is termed as ion beam induced epitaxial crystallization (IBIEC).
Ion beam induced epitaxial crystallization (IBIEC) has been shown to take place in silicon and other materials at considerably lower target temperatures than necessary for thermal annealing when performed under irradiation. Skorupa et al. showed that amorphous Si layer deposited by chemical vapour deposition on silicon substrate can be epitaxially recrystallized by IBIEC at 400°C by implanting Si ions at 330 keV with a dose of $\sim 1 \times 10^{17}$ cm$^{-2}$ [3].

IBIEC provides a way not only to anneal the defects at low processing temperature but has unique characteristics such as layer-by-layer and selective area crystallization, and dynamic defect annealing. Ion energy is chosen such that their projected range is well beyond the original amorphous/crystalline interface [4].

Heera et al. [5] showed that the ion irradiation substantially reduces the onset temperature of both the epitaxial layer regrowth and the random nucleation of crystalline grains.

Low-to-medium energy IBIEC has been mainly ascribed to the migration and recombination of defects (at the amorphous/crystalline (a/c) interface) created by the elastic collisions between ions and target atoms. The role of inelastic scattering processes on IBIEC was first pointed out by Nakata [6]. The inelastic electronic scattering of $^{84}$Kr at energies of 0.5-5 MeV and $^{131,132}$Xe ions at energies of 1-5 MeV were used for IBIEC of amorphous Si layers on crystalline Si substrate at 310-450°C. It was found that the crystallization rate per unit vacancy (normalized crystallization rate) created by the elastic nuclear scattering of the incident ion beam at the amorphous-crystalline (a/c) interface is increased 40-50% by increasing the inelastic electronic scattering three to four fold while maintaining the same elastic nuclear scattering conditions at the a/c interface [7]. Sahoo et al. [8] comprehensively showed 100 MeV Ag ion induced recrystallization of Si, where inelastic scattering process plays a major role.

**Swift Heavy Ion Beam Induced Epitaxial Crystallization (SHIBIEC)**

Epitaxial recrystallization of 200 nm amorphous Si layers by swift heavy ions (50 and 100 MeV Au$^{8+}$) was investigated by Rutherford backscattering spectrometry and Micro-Raman spectroscopy [9]. Good epitaxial recrystallization was observed in the range of 473-673 K, which is a lower temperature regime as compared to the one needed for conventional solid phase epitaxial growth of Si. The self ion (50 MeV Si) induced SHIBIEC showed that the regrowth rate is higher as compared to 50 MeV or 100 MeV Au ions. The enhancement in the regrowth rates shows a systematic dependence on the $S_e/S_n$ ratio. For 50 MeV Si ions, for which the $S_e/S_n$ ratio is about 3000, an enhancement of an order of magnitude in the normalized regrowth rate is seen. The mechanism of layer-by-layer growth of the amorphous layer involves creation and migration of vacancies towards interface causing creation of large
vacant spaces around the interface. These vacant spaces make the thermal vibrations of Si atoms around the interface more free, as a consequence of which, the redistribution and recrystallization of Si atoms occurs resulting in the layer-by-layer epitaxial growth of the amorphous region. For swift heavy ions a hot region around the ion track is created due to the very large electronic energy loss. Vacancies created in this hot region migrate, increase the vacant spaces at the a/c interface, and enhance the regrowth rate dramatically.

SHI induced recrystallization of materials has been studied extensively by Som et al. [10-12]. SHIBIEC of a buried Si₃N₄ layer was observed at temperatures as low as 373 K, at energies where the projectile ions lose their energy mainly by inelastic collision processes [13, 14]. Complete recrystallization of silicon nitride layer, having good quality interfaces with the top- and the substrate-Si, can be obtained this way at significantly lower temperatures of 373, 423 and 473 K for O, Si and Ag ions, respectively.

Recent advances in the scaling down approach of microelectronic devices have given rise to the possibility of using a combination of high-k dielectric materials with high-mobility substrates. Germanium has higher carrier mobilities than silicon (3800 versus 1900 cm² V⁻¹ s⁻¹ for electrons and 1820 versus 500 cm² V⁻¹ s⁻¹ for holes) and is thus attractive material.

Amorphous regions in Ge, produced by ion implantation, regrow epitaxially in the solid phase well below its melting point. The regrowth related defects are very stable ones in high-energy implants in Ge and can be removed only by annealing at temperatures as high as 1123 K [15].

Benyagoub et al. investigated SHIBIEC by irradiating SiC with low and high energy ions and also successively with both types of ions. Sequential irradiations revealed that the damage formed by the low energy ion irradiation can be readily removed by electronic excitations generated by SHI [16].

Som et al. [17] made extensive measurements to get the experimental evidence of intense electronic excitation induced athermal crystallization of a-Ge grown on crystalline Ge substrate by using 100 MeV silver ions to the fluence of 1 × 10¹⁴ ions cm⁻². High-resolution transmission electron microscopic (HRTEM) studies showed complete recrystallization of the a-Ge layer induced by the Ag ions at room temperature. Cross-sectional TEM (XTEM) images collected from various parts of the sample showed uniform morphology, which is single crystalline in nature. This was further confirmed by the selected area electron diffraction (SAED) patterns recorded at different regions in the sample. The observed recrystallization results from the local transient melting due to intense electronic excitation along the ion trajectory and is not because of bulk heating of the sample by the ions. However, no signature of recrystallization in a-Ge was observed when the samples were irradiated by 70 MeV Si and 100 MeV O ions. This indicates to a possible existence of a threshold $S_e$ value for SHI induced recrystallization to take place in a-Ge. Thus, it is clear that room temperature recrystallization of Group IV semiconducting materials is possible only by using swift heavy ions where high electronic excitation induced
processes dominate. The irradiation by 150 MeV Ag ions induce recrystallization in buried Si$_3$N$_4$ and Si overlayer [18].

Results of various experiments in different conditions reveal that although the phenomenon of SHIBIEC resembles the process of IBIEC, it differs from it significantly. High recrystallization rate at low temperature can be accounted for by a mechanism [19] based on the melting of the amorphous zoned through a thermal spike process followed by an epitaxial recrystallization. The conventional IBIEC is triggered by the atomic displacements generated by nuclear collisions, while the effects in SHIIEC is related to the energy deposited by the incoming ions into the target electrons leading to thermal spike process.

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Sputtering

An energetic ion penetrating a solid causes electronic excitations and nuclear collisions. Atoms are emitted from the surface if the energy transfer in such collisions is enough to overcome surface binding energy. This emission process of surface atoms by energetic ions is called sputtering. The average number of atoms ejected from the target per incident ion is called the sputter yield which depends on the ion incident angle, the energy of the ion, the masses of the ion and target atoms, and the surface binding energy of atoms in the target. For a crystalline target the orientation of the crystal axes with respect to the target surface are relevant. Depending on projectile energy loss, different scenario of sputtering occur, that include materials removal due to atomic collision cascade due to nuclear energy loss, so-called “Nuclear Sputtering” and “Electronic Sputtering” governed by the electronic energy loss process. Nuclear sputtering and electronic sputtering are completely different processes. Some ion-induced phenomena depend on the internal (potential) projectile energy, particularly if this potential energy greatly exceeds the kinetic projectile energy. The stored potential energy in a highly charged ion (HCI) can be quite high and produce sputtering in insulators, which is known as the “Potential Sputtering”.

Nuclear Sputtering

The nuclear sputtering is essentially kinetic sputtering, which is due to elastic energy transfer from the ion to solid atom [1]. Any ion impinging on the solid matrix knocks out atoms due to a series of collisions in its journey. Knocked atoms may also remove other atoms from their lattice site due to secondary collisions. Thus a collision cascade develops inside the lattice matrix. When such cascades recoil and reach the target surface with an energy above the surface binding energy, an atom can be ejected. If the target is thin on an atomic scale the collision cascade can reach the back side of the target and atoms can escape the surface providing sputtering ‘in transmission’ mode, if the energy exceeds the surface binding energy.
Nuclear sputtering has a lot of applications in different fields such as: surface cleaning and thinning, thin film deposition, micromachining etc. The controlled removal of material on an atomic scale from the surface by sputtering is the basis of many analytical techniques which provide techniques to determine the sample composition as a function of depth.

**Potential Sputtering**

Some ion-induced phenomena depend on the internal (potential) projectile energy, particularly if this potential energy greatly exceeds the kinetic energy of the projectile. The stored potential energy in a highly charged ion can be quite high. The stored potential energy is equal to the energy spent in removing a part, say \( q \), of their \( Z \) electrons (\( Z \) being the projectiles nuclear charge). This potential energy becomes very large for high values of \( q \) (the ion charge state). Upon surface impact this potential energy induces various inelastic processes while the ion regains its missing \( q \) electrons to again become fully neutralized. The ion deposits its potential energy in a short time (typically about 100 fs) within a small area (typically less than 1 nm\(^2\)). In the course of the neutralization of the highly charged ion (HCI) at the surface a multiply-excited neutral particle with empty inner shells is formed, which is known as the “Hollow Atom”.

Hollow atoms are short-lived multiply-excited neutral atoms which carry a large part of their \( Z \) electrons (\( Z \) is projectile nuclear charge) in high-\( n \) levels while inner shells remain (transiently) empty. This population inversion exists for typically 100 femtoseconds during the interaction of a slow highly charged ion (HCI) with a solid surface. For impact on insulator surfaces the potential energy contained by hollow atom may also cause the release of target atoms and ions via potential sputtering [2-5] which can cause the formation of nanostructures on a surface. For metal surfaces, even rather sudden perturbations of the electronic structure can occur without inducing any structural modification while the excitation energy is being rapidly dissipated in the target material.

The extent to which the electronic relaxation of hollow atom takes place above or below the surface is closely related to the way it dissipates its large potential energy. Emission of electrons and X-ray photons carries away a fraction of the total potential energy originally stored in the highly charged ion. The remaining part is deposited into the solid and converted into electronic excitation of a small surface region. This electronic excitation causes modifications in the material if it is insulating. For metal surfaces the excitation energy gets rapidly dissipated in the target material without inducing any structural modification. On certain insulator surfaces a quite dramatic increase of the yields for total sputtering and secondary ion emission with increasing \( q \) has been observed.
Electronic Sputtering

The ejection of atoms due to the electronic energy loss is referred as the electronic sputtering. It is caused by very high-energy or highly charged heavy ions which lose energy to the solid mostly by electronic stopping power. Simple and complex molecules can be ejected intact into the vapour phase when a material is electronically excited by incident particles, which provides an excellent probe to study the behaviour of condensed matter at high excitation densities and has applications in fields as diverse as astrophysics and biomolecular mass spectrometry [6].

Dependence of Electronic Sputtering on the Film Thickness

Electronically mediated sputtering in thin gold films with 200 MeV Ag ions was studied with ex-situ thickness measurements of the film using X-ray reflectivity technique. The sputter yield was observed to be a few orders of magnitude higher as compared to that normally encountered in the regime of elastic collisions and depends upon the film thickness as shown in Fig. A2.1 [7]. The increased number of oscillations in the irradiated sample is indication of decreased thickness as compared to the unirradiated sample. Sputtering is accompanied by a significant smoothening of the film surface and smearing of the boundaries between the grains. A systematic decrease in sputtering yield of carbon with increase in film (C_{60}/silicon) thickness was observed [8].

Figure A2.1: The patterns of X-ray specular reflectivity of Au films of 15 nm before and after irradiation by 200 MeV Ag ions with fluence of $1 \times 10^{13}$ ions cm$^{-2}$. [Reprinted from A. Gupta and D.K. Avasthi, Phys. Rev., B 64 (2001) 155407, copyright (2009), American Institute of Physics.]
The dependence of electronic sputtering yield on the film thickness was studied in detail using LiF thin films of different thickness (10, 20, 40, 80, 160 and 265 nm) as shown in Fig. A2.2 [9]. It was found that the electronic sputtering decreases with increase in the film thickness as was observed in case of thin Au film. Two distinct regimes of thickness dependence of electronic sputtering were observed. In the regime I, up to 50 nm thickness film, the yield was of the order of $10^6$ atoms/ion, whereas beyond this thickness the yield was an order less ($10^5$ atoms/ion). It was observed that the yield is more sensitive to thickness in regime I than in regime II. High sputtering yield in regime I is due to the combined effect of reduced thickness and grain size, whereas the change in yield in regime II is due to the change in grain size. Also the rate of change of the yield is higher in regime I as compared to regime II. For the dependence of grain size with film thickness, two distinct behaviours were also observed which indicates effective role of grain size on the sputtering, as discussed earlier.

A series of experiments were performed on CaF$_2$ and BaF$_2$ and results were compared with LiF thin films deposited on different substrates (glass, fused silica and Si) [10]. The film thicknesses were 10 and 100 nm for LiF and 100 nm for BaF$_2$ and CaF$_2$ films. The Li/F sputter yields, calculated from ERDA areal concentration versus fluence curves, were $2.3 \times 10^6$ and $6.2 \times 10^4$ atoms/ion from 10 and 100 nm LiF films deposited on Si substrates, whereas they were $7.4 \times 10^6$ and $1.9 \times 10^5$ for 10 and 100 nm LiF films deposited on glass.

**Figure A2.2:** Two different regimes of sputtering as well as grain size vs film thickness are shown. Sputtering yield decreases and grain size of pristine film increases with increasing film thickness. The sputtering is enhanced about one order of magnitude for film <100 nm. [Reprinted from Manvendra Kumar, S.A. Khan, Parasmani Rajput, F. Singh, A. Tripathi, D.K. Avasthi and A.C. Pandey, *J. Appl. Phys.*, **102** (2007) 083510, copyright (2009), American Institute of Physics.]
substrates. The F and Ca sputter yields were $5.3 \times 10^4$ and $2.5 \times 10^4$ atoms/ion for CaF$_2$ deposited on Si substrates and $1.7 \times 10^4$ and $8.1 \times 10^3$ for film on glass substrate. The F and Ba sputter yields are $2.5 \times 10^4$ and $1.2 \times 10^4$ atoms/ion in BaF$_2$ films deposited on Si substrates, while they were $8.3 \times 10^3$ and $4.1 \times 10^3$ atoms/ion for films deposited on glass substrates, respectively. No significant difference in the sputtering yield was observed for films on glass and fused silica substrates. The sputtering yield for film deposited on glass substrate is nearly three times higher than that on Si substrate for all the materials. The observed yields for different materials were compared as a function of band gap of the bulk materials. The band gap of bulk LiF, CaF$_2$ and BaF$_2$ are 14.2, 12.1 and 9.2 eV, respectively. It is clear that the yield increases as the band gap of the material increases.

Insulating materials grown in form of thin films show higher sputtering than that in the corresponding bulk. In thin films, the sputtering has strong dependence on film growth and irradiation parameters. These parameters (grain size and thickness of the film, substrate, irradiation species, energy and temperature) greatly influence the rate of removal of material. The novelty of these experiments is to use equilibrium charge state of the ions for sputtering. For SHI induced surface and/or near-surface modifications of materials, the charge state of the incident ions should be equilibrium charge state i.e. of the order of effective charge defined in the electronic energy loss theory. To reach equilibrium charge state, all the projectiles from the accelerator having +9 charge states were passed through a thin carbon foil. The charge state +25, being the most probable in charge state distribution, was selected by dipole magnet for the experiment. The energy losses of these ions were 15.4, 15.8 and 16.5 keV/nm in LiF, BaF$_2$ and CaF$_2$ crystals, respectively, and contribution of nuclear energy loss is only up to 0.4%. During irradiation the layer thickness and stoichiometry were continuously monitored by ERDA in reflection geometry using a large area position sensitive detector telescope (LAPSDT) [11].

The on-line ERDA analysis performed to measure electronic sputtering of thin films by measuring the loss of material with the fluence and to quantify interface modification by quantifying the changes at the interface with fluence showed that the observations in electronic sputtering were qualitatively explainable by the thermal spike model, and that interface modification was quantitatively explained to be due to the diffusion of species during the transient melt phase. Diffusivity so obtained in the measurements is in the range of $10^{-8}$ to $10^{-6}$ m$^2$ s$^{-1}$. Such a high diffusivity is possible only for the molten state [12].

**Dependence of Electronic Sputtering on Ion Velocity, Charge State and Substrate**

Ion velocity, charge state and substrate dependence of electronic sputtering of fullerene (C$_{60}$) was studied with thin films deposited on Si and glass substrates.
using Au and Ag ions of different energies. Slower ion having same electronic energy deposition ($S_e$) as compared to its high velocity counterpart erodes more. $C_{60}$ films deposited on more insulating substrate shows higher sputtering yield as compared to those deposited on Si substrate. No charge state effect was observed in the electronic sputtering yield within the experimental error of the set up [13].

**Structural Effects on Electronic Sputtering**

Structural effect on electronic sputtering of hydrogenated amorphous carbon films on bombardment with 150 MeV Ag$^{13+}$ ions were studied with on-line elastic recoil detection analysis (ERDA) technique. A large erosion ($\sim 10^5$ atoms/ion) of C and H from hydrogenated amorphous carbon films (a-C:H) was observed [14].

**Angular Distribution of the Sputtering Yield**

The angular distribution of the sputtering yield from highly oriented pyrolytic graphite sample irradiated with a 130 MeV Ag beam was studied using a high resolution ERDA set up. The maximum sputtering yield is observed at 53°, falling rapidly to almost zero at 90°, with an average sputter yield of $5.5 \times 10^5$ atoms/ion [15].

Electronic sputtering produces high sputtering yields from insulators, as the electronic excitations that cause sputtering are not immediately quenched, as they would be in a conductor. The yield of the electronically sputtered atoms is higher normal to the sample surface and as a function of the electronic energy loss ($S_e$) of the projectiles, the total sputter yield follows a $S_e^4$ law [16].

The angular dependence of electronic sputtering from HOPG with 120 MeV Au ion beam for three cases: from crystalline highly oriented pyrolytic graphite (HOPG) for (A) normal and (B) 70° incidence and from (C) amorphous carbon sample for normal incidence was studied in detail [17]. The sputtering yield shows an anisotropic distribution for all the three cases studied. However, the anomalous peak observed at 53° for normal incidence for HOPG sample is found to shift to 73° when the sample is tilted by 20°. Similar study with amorphous carbon sample shows no peak. The peaks observed in sputtering yield distribution have been attributed to the crystalline structure of the sample which allows the preferential release of pressure pulse along the crystal axis. The high exponent of over-cosine distribution ($n = 3.2–3.8$) signifies formation of intense pressure pulse induced jet-like sputtering.

**Importance of Nuclear Energy Loss in Electronic Sputtering**

Effect of 100 MeV Au irradiation on embedded Au nanoclusters in silica glass was analyzed using Rutherford backscattering spectrometry, transmission electron microscopy and optical absorption spectroscopy [18]. At lower irradiation fluence the high energy heavy ion irradiation has been found to result in a loss in Au due to an outward movement of the NCs together with a
growth in size. At the highest irradiation fluence, almost 80% Au was lost, with only a few large NCs seen which had moved to the surface. These were found to be of a deformed non-spherical shape. The amount of Au lost was found to increase linearly with irradiation fluence indicating the movement of Au to be not dominated by diffusion. The enhanced sputtering of Au under SHI irradiation has been suggested to be due to collision cascades produced by nuclear energy loss \( S_n \) operating along with the inelastic scattering due to \( S_e \) [19].

**Surface Modification by Electronic Sputtering**

Swift heavy ions induced effects on optical (colour centres), structural and surface (electronic sputtering and morphology) modifications, in nano-grains LiF thin films were studied by glancing angle X-ray diffraction, optical absorption, photoluminescence and elastic recoil detection analysis techniques. Results show that grain size and irradiation temperature play a crucial role in materials modifications as a function of fluence for the selected ion beam parameters. Also for the first time, lamellae formation was observed in LiF thin films after a high fluence irradiation of \( 5 \times 10^{13} \) ions cm\(^{-2} \) at liquid nitrogen temperature with 120 MeV Ag ions irradiation under grazing incidence (~10°) [20].

**Surface Structuring through the Electronic Sputtering**

Structure dependence of electronic sputtering of a-C:H films by 80 MeV Ni\(^{8+} \) and 150 MeV Ag\(^{13+} \) ion irradiations were analyzed from the characteristic graphitic (G) and disordered (D) modes of Raman vibration [21].

Scanning force microscopy studies on the organic single crystals irradiated with GeV energy ions reveal two different defect morphologies, i.e., either hillocks or craters. The defect morphology depends exclusively on the electronic energy loss of ions. For the same crystal namely, benzoyl glycine, hillocks are produced at an energy loss less than 4 keV/nm, and above this value up to 15 keV/nm craters are produced [22].

Formation of nanoscale metallic structures by the impact of 200 MeV Au\(^{15+} \) ions on cupric nitride thin film surface was studied with on-line elastic recoil detection analysis (ERDA) technique. A large depletion of N (~75% depletion) from the films due to electronic sputtering effect was observed whereas the copper content remains unchanged. The surface of the pristine film studied by atomic force microscope (AFM) shows nanodimensional grain formation. Conducting AFM (CAFM) measurements show that at certain regions (10–30 nm) of the irradiated film surface a rapid rise of current (~9000 pA) takes place. Enhancement of electron emission together with conducting AFM measurements lead to the conclusion that conductivity of the surface enhances due to formation of nanodimensional metallic zones under Au ion impact [23].
Electronic sputtering of different allotropes of carbon (diamond, graphite, fullerene, a-C and a-C:H) were studied under 200 MeV Au$^{15+}$ ion irradiation. Erosion behaviour is distinctly different in different allotropes as observed by ERDA. Hardest known material diamond does not show any sputtering within the detection limit of the experimental set up, whereas the soft polymers like a-C:H shows highest sputtering yield ($5.8 \times 10^5$ atoms/ion). Yields in case of other allotropes are $1 \times 10^3$ atoms/ion (graphite), $3 \times 10^4$ atoms/ion (fullerene), $1.8 \times 10^4$ atoms/ion (a-C), respectively [24].

Energy dependent sputtering of nanoclusters from a nanodisperse target of Au nanoparticles, prepared on Si substrate was studied at different ion energies to analyze the synergetic effects of nuclear stopping and electronic stopping [25].

Highly oriented pyrolytic graphite (HOPG) samples (Grade ZYB with grain size ~1 mm) were studied with 150 MeV Au beam using scanning tunnelling microscopy (STM) and scanning tunnelling spectroscopy (STS). The formation of hillocks is observed for the samples irradiated with fluences of $1 \times 10^{11}$ ions cm$^{-2}$, $1 \times 10^{12}$ ions cm$^{-2}$ and $1 \times 10^{13}$ ions cm$^{-2}$ with typical diameters of 6.2, 2.2 and 1.5 nm, respectively. No hillocks are observed for the sample irradiated with fluence of $2 \times 10^{13}$ ions cm$^{-2}$, though the formations of small craters were observed. The formation of hillocks is attributed to nuclear energy loss induced collision cascades near the surface. The reduction in hillocks size and formation of craters at higher fluence is attributed to the electronic sputtering from the surface. The STS studies of I–V characteristics show an increasing ohmic behaviour with fluence which is attributed to increasing metallic state for HOPG surface due to irradiation induced increase of carbon bond lengths [26].

Ejection of ZnS nanoparticles from ZnS film on Au irradiation was studied using transmission electron microscopy. No nanoparticle (NP) could be observed on irradiation with 35 keV Au ions. However, 2–7 nm size NPs were observed on MeV irradiations at room temperature. For particle sizes ≥ 3 nm, the distributions could be fitted to power law with decay exponents varying between 2 and 3.5. At 2 MeV, after correction for cluster breakup effects, the decay exponent was found to be close to 2, indicating shock waves induced ejection to be the dominant mechanism. The corrected decay exponent for the 100 MeV Au irradiation case was found to be about 2.6 [27].

The study of the influence of grain size on electronic sputtering of LiF thin showed a reduction in sputter yield with increasing grain size. The electrons liberated in different directions from the ion track have different diffusion length according to its energy. The motion of the electrons is affected by the smaller grain size due to grain boundary scattering resulting in reduction in the mean diffusion length of the electrons, which finally enhanced energy deposition inside the grains and thus the sputter yield. From ERDA measurements, reduction in the area concentration of F and Li due to ion bombardment with fluence was observed indicating the sputtering of Li and F from the film. It
was found that the Li and F are nearly equally present in the film before and after sputtering, which showed the stoichiometric sputtering of LiF. The grain size decreased from 58 nm to 22 nm with the decrease in the substrate temperature from 500 K to 77 K, respectively, while the film thickness (150 nm) was kept constant for all the depositions. The reduction in sputter yield, from $\sim 5.5 \times 10^4$ to $7.1 \times 10^3$ atoms/ ion, was observed with the increase in grain size of the film [28]. The sputtering from LiF single crystal were measured by Toulemonde et al. [29] using catcher technique and the yield of Li and F was found to be about 17530 and 15790 atoms/ion, respectively, for ions having electronic energy loss of 16.4 keV/nm.

Comparing the observed yield obtained in the films having different grain sizes, it was observed that the yield ($1.6 \times 10^4$ atoms/ion for F) in case of the film having the grains of 46.8 nm is comparable to the existing results, while in other cases there is huge difference in the sputter yield with change in the grain size of the films.

The experimental results are assessed within the framework of thermal spike model along with size effect in thin films and influence of substrate. Reduction in the film thickness and the grain size can restrict the motion of the excited electrons because of the scattering from surface and interface and grain boundaries, respectively. So, the size effect can result in reduction of the mean diffusion length, $\lambda$, of the electrons resulting in increase in the deposition of energy in confined region, which finally can enhance the temperature spike in the thinner films/films having smaller grains. On the other hand, in smaller grains and thinner films, the duration of thermal spike will be more because of less out-diffusion. As a quantitative approach to explain the higher sputtering yield in case of insulator substrate, inelastic thermal spike model can be applied. As the range of ion (>15 µm for all the cases) is more than the film thickness, a thermal spike will be developed in the substrate also. This temperature spike can increase the temperature generated in the film resulting in higher sputtering. The temperature in Si substrate will smear out more efficiently due to its higher thermal conductivity (nearly 40 times more than that of glass/fused silica) and yield will be higher for glass/fused silica substrates. On the other hand, because of amorphous nature of glass/fused silica, the electron-phonon coupling strength will be stronger than that in Si, resulting in higher temperature rise in glass and silica substrates supported by thermal spike model. The contribution from temperature developed in substrate will enhance the yield and the sputtering will be lower in case of the film deposited on Si substrate than that for glass/fused silica.

Two exponents $\delta$ for the size distribution of $n$-atom clusters, $Y(n) \sim n^{-\delta}$, were found in Au clusters sputtered from embedded Au nanoparticles under swift heavy ion irradiation [30]. For small clusters, below 12.5 nm in size, $\delta$ was found to be 3/2, which can be due to a steady state aggregation process with size independent aggregation. For larger clusters, a $\delta$ value of 7/2 is suggested, which might come from a dynamical transition to another steady
state where aggregation and evaporation rates are size dependent. In the present case, the observed decay exponents do not support any possibility of a thermodynamic liquid-gas-type phase transition taking place, resulting in cluster formation. The results imply that observables such as the sputtering yield may be used as signatures of the fast electron-lattice energy transfer in the electronic energy-loss regime [31].

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Applications of the PIXE Technique

Proton Induced X-Ray Emission (PIXE), non-destructive elemental analysis technique, has proved to be of immense importance in diverse areas such as forensic science, bio-medical field, archaeological and environmental studies, geochemical prospecting etc.

Normally PIXE samples are mounted inside a vacuum chamber and exposed to the proton beam for measurements. It creates problems for archeological large samples and biological targets. It is possible to extract the beam outside the accelerator beam line and plan measurements at atmospheric pressure with appropriate modifications, i.e. PIXE with External Beam.

Availability of micro-focussed proton beam has opened up a new horizon for exploiting PIXE technique in many disciplines. PIXE using micro-focussed beams, called Micro-PIXE, gives additional capability of microscopic analysis.

Trace Elements in Water

Elemental composition of water is of great importance as it is the prime source of the trace elements essential for the growth of living organisms. A systematic study of river water samples collected from various places in the Indo Gangetic valley (River Ganges) was done. The trace levels of several elements increase as one goes down the stream and pass through cities [1]. Concentrations of certain heavy elements are large near industrial areas and decrease downstream [2]. Samples from a hot spring, well known for its curative properties for skin diseases contain large S content, a main constituent of several skin medications. A comparison of the trace levels in various mineral water samples was carried out by Kennedy et al. [3].

A study of the drinking water from Salt Lake City, a residential locality in Kolkata, India, was done using a chelating agent (NaDDTC) for the pre-concentration of the trace elements. A large number of elements, namely Ca, Ti, Mn, Fe, Co, Ni, Cu, Zn, As, Sr, Ba, Tl and Pb were analyzed [4]. In a similar study at Chandigarh [5], the main pollutants in the river samples around Patiala District in Punjab were found to be K, Ca and S with the relative percentage of K as maximum.
Bolormaa et al. [6] investigated the effect of mining activity on the environment by studying the heavy metal contents in the Boroo River water samples collected in mining area of Mongolia. Similarly, Saleh [7] carried out investigations on trace metal contamination in drinking water in Jordan. Quinones et al. [8] used the PIXE technique with the objective of characterizing and monitoring of the trace elements in the water of São Francisco River, as well as to provide valuable information about the levels of metallic ions pollutants.

**Analysis of Human Hair Samples**

Hair is the most easily accessible biological tissue. It contains trace elements which reflect metabolic changes in the body over long periods of time. Also, continuous exposure to environment leaves an imprint of atmospheric pollutants. The elemental composition of hair vary greatly from individual to individual and with geographical location. Varier et al. [9] studied whether the elemental content exhibits any systematic pattern for members of the same family and for different families in comparable environment and nutritional background. Hair samples from the different parts of the head of the same person and from different persons were subjected to PIXE analysis. The standard deviations in the estimated trace levels were about 17-28% in samples from same person and about 30% to 69% in samples from different individuals.

It was found that the zinc content remains constant along the length of the hair and is not governed by external pollutants but by internal metabolism. Concentration ratios of all elements in the samples relative to Zn were extracted from the data. Following are a few results from the study:

- Mn/Zn and Fe/Zn ratios are higher in the family from rural areas.
- Females were found to have higher Cu/Zn ratio.
- Married females were found to have higher Pb/Zn ratio.

No definite trend in the copper content of black hair (blackness due to a copper containing pigment called melanin) versus grey hair is found. The reason may be that other sources of copper content may mask the copper in melanin. It was observed that married women have more lead in their hair, probably due to the *kumkum* they apply on their foreheads.

Small scale miners in the mountainous regions of Benguet Province in the Philippines extract gold using a method which involves the use of mercury, via amalgamation. In the separation of gold from mercury the method involves the release of mercury vapour into the atmosphere. This is therefore expected to affect the people living in the nearby areas, which was investigated. This study involves the accumulation of baseline data on the extent of mercury contamination in humans through the analysis of their hair [10]. In 1989, Hursh et al. [11] studied human volunteers and found that uptake of mercury vapour through the skin is only about 1% of the uptake through inhalation [12]. In this light, any residual mercury which might have deposited in human hair is speculated to give an indication of how much mercury vapour the subject could
have actually inhaled. A high concentration of mercury in the sample can therefore be indicative of the high rate of intake of the mercury vapour through inhalation.

Arsenic pollution in Bangladesh and Japan was studied using hair samples by Habib et al. [13]. The results show markedly higher levels of arsenic, manganese, iron and lead where the later three elements show a positive relation with arsenic in the case of Bangladeshi as compared to the samples from Japan. On the other hand, selenium concentrations show very low level in the Bangladeshi samples compared to Japanese, displaying an inverse relationship with arsenic. Sera et al. did quantitative analysis of untreated hair samples for monitoring human exposure to heavy metals. It was found that the concentration of mercury and arsenic in hairs taken from different parts of a body does not show significant difference, demonstrating that the concentration in hairs are good index for an estimation of human exposure to these toxic elements [14].

**Forensic Science Studies**

Elemental analysis has been found to be quite helpful in the identification of crime related samples, with possibility of identifying the criminal. For example, in cases of gunshot firing, the type of bullet and distance of firing are important parameters. The bullet carries along with it a part of the primer, gun powder and also a part of the material of the gun itself. Some of these get deposited around the bullet hole on the body of the victim. An analysis of the radial distribution of these elements (Sb, Ba, Cu, Pb and Fe) can be helpful in this respect. Laboratory simulated samples analysed for the gunshot residues by the PIXE technique shows clearly the dependence of the radial distributions of the various elements on the distance of firing [15]. Representative results for Pb are shown in Fig. A3.1.

![Figure A3.1](image.png)

**Figure A3.1:** Relative intensities of Pb detected as a function of distance from the centre of bullet hole. Each curve corresponds to a different firing distance as specified. [Reprinted from S. Sen, K.M. Varier, G.K. Mehta, M.S. Rao, P. Sen and N. Panigrahi, *Nucl. Instr. and Meth.*, **181** (1981) 517 with permission from Elsevier.]
The non-destructive characteristic of PIXE has proven to be extremely valuable in the area of forensic applications. Samples can be analyzed maintaining their integrity. For example, bone samples from a victim in order to determine the presence of lead and samples returned to the court as evidence.

Warren et al. [16] used PIXE analysis in two forensic contexts: (1) case of cremation in which the nature of the remains is questioned and (2) cases of death by gunshot wound. In the first case, elemental analysis by PIXE revealed that the purported cremated remains are not bone, and in the second that radiopaque metallic residue embedded in bone is composed of lead from a projectile.

Other crime related samples subjected to the PIXE analysis include typewritten papers, nails, hair, blood stains etc. [17].

**Geochemical Prospecting**

Geochemical studies are largely concerned with establishing the distribution patterns of elements or group of elements, and classifying the laws governing these distribution patterns in natural systems. Mineral deposits represent anomalous concentrations of specific elements, usually within a relatively confined volume of the Earth’s crust. Most mineral deposits include a central zone, or core, in which the valuable elements or minerals are concentrated, often in percentage quantities, to a degree sufficient to permit economic exploitation. The valuable elements surrounding this core generally decrease in concentration until they reach levels, measured in parts per million (ppm) or parts per billion (ppb), which appreciably exceed the normal background level of the enclosing rocks. These zones or halos afford means by which mineral deposits can be detected and traced; they are the geochemical anomalies being sought by all geochemical prospectors. The use of trace elements as indicators of geochemical processes provides an excellent way.

A large number of samples of the U.P. State Geology & Mining Department in India from eight regions spread over several kilometres in Kumaun Hills were analyzed [18]. It was concluded that Koirali region has good prospect for tungsten mining and indicated presence of uranium in Kuria Hill in Mirzapur district. A few samples from Orissa Mining Corporation indicated presence of Pt and Mo in the specimens from Sukinda Valley.

Ahmed [19] analyzed geochemical samples by the micro-PIXE technique of gold-bearing rocks, phosphorite ores and volcanic sediments. Elemental composition and distribution maps across single mineral grains, fluid inclusions, grain boundaries and matrices were measured. Frietas et al. [20] studied the serpentinophytes from north-east of Portugal for trace metal accumulation and to study its relevance to the management of mine environment. Ryan et al. [21, 22] carried out quantitative PIXE microanalysis of geological material using the CSIRO proton microprobe.
Archaeological Studies

Ancient cultures had developed highly sophisticated techniques manifested in beautiful artworks. For example pigments used in mural paintings provide very significant information. Ancient Indian coins made of Cu and Ag of Hindu Sahi dynasty (990–1015 AD) were analyzed. The presence of trace elements like Ti, Cr, Ni, Fe and Pb provides information about the source of Cu as from Khetri mines in Rajasthan. Analysis of metallic compositions of coins can provide valuable information regarding coin minting, methodology, provenance, art, culture and economics of the minting time [23, 24].

External proton induced X-ray emission (PIXE) is a good scientific method for nondestructive analysis of coins preserved in museum. Ten Kushana copper coins (3rd and 4th century AD) from the Orissa State Museum, Bhubaneswar were analyzed by using an external beam PIXE [25]. The study reveals that copper is the main constituent of the coins, with minor/trace elements like titanium, iron, nickel, zinc, lead and bismuth.

A quantitative analysis of an ancient statue was performed by external beam proton induced X-ray emission for the purpose of identifying its originality. The elemental composition of the statue is compared with that of several samples with definite ages. The analysed elements were Fe, Cu, Ag, Au and Hg for gold coating and Fe, Ni, Cu, Zn, As, Ag, Sn, Au, Pb and Bi for bronze body [26-28].

Sanchez del Reo et al. [29] applied the PIXE technique to the analysis of blue pigments contained in several Mesoamerican mural samples. Problems concerning the determination of technology and provenance of archaeological metals have been studied by PIXE and XRF by M.F. Guerra [30]. Bugoi et al. [31] have utilized a micro-PIXE set up for the study of the metal provenness of gold archaeological samples.

Bio-Medical Applications

The importance of trace elements in biomedical processes has been recognized but the details in which it manifests their biological role needs detailed study. Blood, tooth, nail, hair etc. have been investigated. However, most of these have been of exploratory type. As an example, blood samples of rheumatoid and non-rheumatoid group patients were analyzed as a part of a student project of the Medical College in Kanpur. It was found that Zn level is relatively high for non-rheumatoid group [32]. A patient lying in coma in the All India Institute of Medical Sciences in New Delhi was suspected of Hg poisoning but no technique was available to test the diagnosis. PIXE analysis done in IIT Kanpur helped the diagnosis and monitoring the progress of the treatment [32].

Quantitative analysis of lead levels in blood is important from the point of monitoring the effect of environment pollution on the human health. PIXE facility in the BARC laboratory in Mumbai was used to determine the blood lead levels in children admitted to hospital in Mumbai with suspected lead
poisoning, ascribable to environmental pollution from heavy vehicular traffic and industrial sources. The lead concentration in the blood samples of the patients varied from 10 to 600 mg/dl.

The samples were grouped on the basis of clinical findings such as hypochromic microcytic anaemia, gastrointestinal symptomology, encephalopathy of unknown actiology, mental retardation and pica. The samples having significantly elevated (> 140 mg/dl) blood lead levels belonged mostly to the patients of encephalopathy, pica and anaemia groups [33].

Trace elemental analysis was carried out in the biological samples of cancer afflicted intestine [34]. It was found that the concentration of the elements Cr, Fe and Ni are higher in the cancerous tissue of the intestine than those observed in the normal tissue, whereas the concentration levels of the element Zn is slightly lower in the cancer tissue of intestine than that observed in the normal tissue. The concentrations of S, Cl, K, Ca, Ti, Mn, Co and Cu in the cancer tissue of the intestine are in agreement with those observed in the normal tissues within standard deviations. The present results support the belief that Ni and Cr are carcinogenic agents. The observed slightly low levels of zinc in the cancer tissue of the intestine suggest that zinc could possibly inhibit the tumour growth and development of neoplastic transformation. Trace elemental correlation studies in human malignant and normal tissues in different parts has been studied using PIXE technique [35-38].

Trace element changes in tissues of experimental animals (rats) as a result of liver necrosis or cirrhosis and the determination of the regional distribution of trace elements in the human brain have been done by Maenhaut et al. [39].

Trace elemental analysis was carried out in the tissue samples of normal, benign hypertrophic and carcinoma prostate using 3 MeV proton beam of Institute of Physics, Bhubaneswar. It was observed that in benign tissues the concentrations of Cl, K, Zn and Se are lower and those of Cr, Fe, Ni and Cu are higher than in normal tissues. The concentrations of K, Ca, Zn, Se and Br are lower and those of Ti, Cr, Mn, Fe, Ni and Cu are significantly higher in cancerous tissues than in normal tissues. Free radicals generated by elevated levels of Cr, Fe, Ni and Cu possibly initiate and promote prostate cancer by oxidative DNA damage. The excess Cu levels in cancerous tissues support the fact that Cu promotes cancer through angiogenesis. The higher levels of Fe observed in cancerous tissues might be a consequence of tumour growth through angiogenesis. Significantly higher levels of Ni and Cr observed in carcinoma tissues support the well established role of Ni and Cr as carcinogens. It is likely that the observed low levels of Zn and Se in cancerous tissues lead to the development of prostate cancer owing to a decrease in antioxidative defense capacity and impaired immune function of cells and also suggest that the inability to retain high levels of Zn and Se may possibly be an important factor in the development and progression of malignant prostate cells [40, 41]. However, in order to substantiate the observed elevated or deficient levels of trace elements in initiating, promoting and inhibiting prostate cancer, several cellular and molecular studies are required.
The role of some trace elements in the formation of gallstones was investigated. It was observed that 14 minor/trace elements, namely S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Br and Pb, were present in the cholesterol stone samples. The average concentration of Fe in south Indian (Chennai region) gallstone samples (503.4 ppm) is about 2.5 times more than that of the east Indian gallstone samples (205.0 ppm), whereas the concentration of Fe is still higher in other parts of south India (848.2 ppm) [42]. The higher concentration of Cu (in some parts of south India except the Chennai region) and Fe in south Indian cholesterol stone samples may be due to the intake of tamarind (Garcinia camboginina) as their regular food. The thermogravimetry curves provided information on the thermal decompositions of cholesterol stones.

The iron content in human alveolar macrophages has been studied by Corhe et al. [43] to investigate the clinical usefulness of the PIXE technique in occupational respiratory medicine and in various pulmonary diseases. Surgically excised malignant and normal tumours of breast tissue were subjected to PIXE investigations by Vatankhah et al. [44]. Statistically significant differences in the trace levels were noted between malignant and normal tumours.

PIXE using micro-focussed beams, called MicroPIXE, gives the additional capability of microscopic analysis. It can, for example, quantify the metal content of protein molecules. It is being utilized, for example, in Columbia University for trying to correlate trace element deficiencies with Alzheimer disease. Trace elements, such as Fe, Mn, Zn and Co, form the active centres of enzyme proteins and play important roles in their biochemical functions. Fluctuations in these elements affect the function of living tissues. Each tissue and organ has different patterns of trace elements. The micro-PIXE system at Tohoku University Japan [45], developed by Ishii’s group, enables to obtain two-dimensional images of tracer elements in tissue slices with high spatial resolution. When combined with microbeam scanning of a sample surface, the PIXE method provides the spatial distribution of the elements in a cell. Two-dimensional maps of elemental compositions can be generated by scanning the microPIXE beam across the target.

REFERENCES


Applications of RBS and ERDA

Introduction
The impingement of ions on solid results in scattering of ion, producing recoil, emission of photons such as X-rays, gamma rays and visible light, emission of gases for polymer targets, etc. All these events are utilized by developing techniques for the characterization of materials. The scattering of incident ion and the complementary event i.e. recoils are used in these techniques. Elastic recoil detection analysis (ERDA) is used for light elements depth profiling and Rutherford backscattering spectrometry for depth profiling of high Z elements. Some examples of these techniques are described here.

Rutherford Backscattering Spectrometry (RBS)

Compositional Analysis
The determination of stoichiometry of thin films is one of the major uses of RBS. It has been used very effectively in the determination of the metal content in metal dielectric nanocomposite thin films [1-5]. One such example is given here [6]. A typical RBS spectrum for the Au-silica nanocomposite thin film (Au nanoparticles embedded in silica) is shown in Fig. A4.1 for the films with two different Au content (10 at% and 20 at%). The peak in higher energy region of scattered $\alpha$ particle represents Au with two different contents. At lower energy, two edges correspond to the Si of the substrate and Si in silica of the nanocomposite thin film. A small peak riding over the plateau region corresponds to oxygen in silica in the nanocomposite thin film. The information of metal content in the noble metal silica nanocomposite thin film is useful in simulating the surface plasmon resonance, where metal content is one of the input parameters for simulation.

Ion Beam Mixing
RBS is one of the most effective tools for the interface analysis for the ion beam mixing experiments [7-18]. One example is discussed here [19].

Figure A4.2: RBS spectra of pristine and SHI (120 MeV Au ions) irradiated sample of Ni deposited on Teflon. Asymmetrical broadening and shift of the low energy edge of Ni peak indicates strong mixing. [Reprinted from Jai Prakash, A. Tripathi, S.A. Khan, J.C. Pivin, F. Singh, Jalal Tripathi, Sarvesh Kumar and D.K. Avasthi, *Vacuum*, 84 (2010) 1275 with permission from Elsevier.]
RBS spectra of the pristine and the irradiated Ni/Teflon system (Ni thin film deposited on Teflon polymer sheet) are shown in Fig. A4.2 [19]. RBS spectrum of the pristine film has a low energy edge which correspond to F present in Teflon, whereas the peak at higher energy corresponds to Au. The RBS spectrum of irradiated sample has F edge extending as tail towards higher energy while the low energy edge of Au extends towards lower energy region which indicate that the irradiated sample had mixed region of Au and Teflon.

When interface in a multilayer is to be analyzed in which the layer thickness is smaller than the resolution (~10 nm) of RBS, one requires high resolution RBS. For example, the interface of SHI C$_{60}$/a-Si multilayer deposited on Si is examined by high resolution RBS using an electrostatic deflector. The high resolution RBS spectra recorded for pristine and SHI irradiated films are shown in Fig. A4.3 [20].

![Figure A4.3](image)

Figure A4.3: High resolution RBS spectra of pristine and SHI irradiated multilayer of C$_{60}$/Si. [Reprinted from S.K. Srivastava, D. Kabiraj, B. Schattat, H.D. Carstanjen and D.K.Avasthi, *Nucl. Instr. and Meth.*, **B 219-220** (2004) 815 with permission from Elsevier.]

**Nuclear/Electronic Sputtering**

The sputtering measurements of thin film are studied very effectively by measuring elemental composition of pristine and irradiated samples using RBS [21, 22]. The RBS spectra of pristine and 100 MeV Ag ion irradiated (at a fluence of $10^{13}$ ions cm$^{-2}$) Au film are shown in Fig. A4.4(a). The metal content clearly decreases with increase of SHI fluence, revealing SHI induced electronic sputtering. The decrease in film thickness with such low fluence is an indication
of large electronic sputtering due to nano dimensional film thickness. RBS spectra for pristine (of the same thickness as in previous case) and 50 keV Si ion irradiated Au film at a fluence of $10^{16}$ ions cm$^{-2}$ are shown in Fig. A4.4(b). The decrease in peak is due to loss of Au because of nuclear sputtering. The same film under SHI irradiation results in decrease in Au film thickness at a fluence of $10^{13}$ ions cm$^{-2}$, showing a large sputtering due to electronic energy loss. Thus electronic and nuclear sputtering are determined by RBS.

Figure A4.4: RBS spectra of the pristine and irradiated Au thin film on glass (a) for SHI with 100 MeV Ag ions and (b) for low energy with 50 keV Si ion.
RBS Channelling

RBS channelling is a unique way to measure strain in strained layer superlattice (SLS). An experiment to study the modification of strain in SHI irradiated heterostructures (InGaAs/GaAs), reveals that the compressive strain decreases with ion fluence without loss of crystallinity [23]. The modification in strain with SHI irradiation results in controlled modification in optical properties. RBS channelling revealed that SHI irradiation can induce strain in an initially lattice matched system and can result in decrease in the compressive strain in an initially strained system without effecting the crystalline/interface quality [24]. Experiments of RBS channelling at room temperature and low temperature are capable of probing the atomic vibrations in lattice. RBS channelling is an effective tool to study ion induced epitaxial crystallization [25].

H Depth Profiling by Conventional ERDA

Correlation of H Content with the Microstructure

Hydrogen contributes to the carbon $sp^3$ or $sp^2$ bonding, which in turn affects the hybridization of carbon-carbon atoms during growth leading to graphitic or diamond-like character. Further, the microstructure of a-C:H films is intimately related to the total H content and the nature of C–C and C–H bonds in the films. ERDA provides an excellent technique for studying the correlation of hydrogen content with the microstructure of a-C:H films [26].

Dependence of Hardness on H Content

The depth profiling hydrogen in thin diamond-like carbon (DLC) films produced using dc glow discharge decomposition of acetylene show that high hardness DLC films have a low hydrogen content and higher thermal stability [27].

Ion Induced Delamination

Delamination of CVD diamond films deposited on Si substrates was studied with MeV He ions. It is found that film gets delaminated during 1.5 MeV He$^+$-ion irradiation. In-situ monitoring of hydrogen, during irradiation, using ERDA provides a way to estimate the threshold ion fluence for exfoliation to occur [28].

H Depth Profiling of Diamond Films

H profiling in diamond film grown by hot filament CVD and microwave CVD for different deposition pressure gave valuable information on the mechanism of growth of diamond films [29].

H Depletion from $KH_2PO_4$ Under He$^+$-ion Bombardment

Hydrogen depletion was probed in the optoelectronically important insulating material, $KH_2PO_4$ (KDP). It was found that depletion of hydrogen occurs under He$^+$-ion bombardment [30]. Investigation of the effect of Au of varying
thicknesses showed that a 6 nm Au layer on KDP acts as a barrier and reduces the depletion of hydrogen from the sample by a considerable amount. These results provide insight into the ion beam induced structural and compositional changes in these materials to tailor their properties.

**Ion Track Radius by H Loss Measurements**

The diameter of the track produced by the ion is a quantity of interest for the understanding of basic ion insulator interaction. There have been attempts to measure the track diameters by scanning force microscopy and other state-of-the-art surface morphology probing equipments. A novel approach was demonstrated by the measurement of H loss during ion irradiation by the online elastic recoil detection analysis [31].

Swift heavy ion irradiation leads to decrease in hydrogen content in hydrogen containing materials. The hydrogen loss can be further correlated with other chemical transformations in these materials by the online-ERD measurements. For example, it was shown that 120 MeV Au irradiation of 400-500 nm thick films of methyltriethosilane on silicon substrates led to hydrogen loss from a narrow cylindrical zone which caused formation of C rich cylindrical zones [32].

**Effect of Incident Ion Charge State on H Loss**

The effect of charge state on hydrogen loss from polypropylene (PP) and polyethylene terephthalate (PET) foils was studied by online-ERD using 130 MeV Ag ions with 11\(^+\), 14\(^+\) and 25\(^+\) charge states (q). It is known that the swift heavy ions with different charge states deposit different amount of energy in the first 10 nm or so while travelling in the material. This small thickness was not resolvable by the detection system and hence top 100 nm were considered for comparison. Hydrogen release cross-section from top 100 nm was found to vary as \(q^n\), \(n\) being 2.98 and 1.94 for PP and PET respectively. The radii of ion tracks formed was also found to depend on the charge state [33].

**H Depth Profiling in Pr-Pd Layers**

The role of ion tracks formed in Pr-Pd layers on an all-round enhancement in the hydrogenation properties of these films was investigated with 120 MeV Ag\(^+\) ions. Same ion beam was used to record hydrogen concentration in hydrogenation (absorption of hydrogen) and dehydrogenation (removal of hydrogen by creating vacuum) processes of these films by ERDA. The hydrogenation property has been found to be strongly influenced by the ion fluence. About 17.8% increase in the hydrogen stoichiometry value during hydrogenation, near maximal removal (about 31%) during dehydrogenation was observed for the highest ion fluence. Non-equilibrium structural changes during ion irradiation leading to the formation of nanotracks throughout the film thickness may provide two-way transport routes for H diffusion. This
study provides a novel methodology of improving the hydrogenation properties of materials [34, 35].

The hydrogenation properties of nanoparticle Gd (grain size ~8 nm) and nanocrystalline Gd samples (grain size ~30 nm) were studied by ERDA [36]. The nanoparticle sample exhibited a larger difference in the stoichiometry ([H]/[Gd]) values (2.9 and 1.7) in comparison to polycrystalline sample (2.4 and 2.0) in the hydrogenated and dehydrogenated states respectively.

**H Depth Profiling in NEG Strip**

Hydrogen depth profiling of Non Evaporable Getter (NEG) material (an alloy of V, Fe and Zr) was performed in transmission geometry [37]. H recoil spectra of pristine and used NEG strip is shown in Fig. A4.5. NEG has the property of absorbing the gases after its activation and is, therefore, of interest in vacuum technology for its use as pumping device. Since hydrogen is one of the most dominant residual gases in vacuum, an experiment was performed to quantify the absorbed H in NEG strip used in vacuum pumping and in the pristine NEG strip.

![Figure A4.5: H recoil spectra of pristine and used (for vacuum pumping application) NEG strip. [Reprinted from D.K. Avasthi, Material Science Forum, 248-249 (1997) 405 with permission from Trans Tech Publications.]](image_url)
Stoichiometric Analysis of a-SiN\textsubscript{x}:H Thin Film by ERDA

Stoichiometric analysis of a thin a-SiN\textsubscript{x}:H film on Fe substrate was performed [38]. A film on Fe substrate instead of Si substrate because of the interest to detect the content of Si along with other elements in the film and the recoils from Fe were stopped in stopper foil [38]. The sample was tilted at an angle of 20° and detector was kept at 34° with a stopper foil of 6 µm in front of it to stop unwanted scattered Ni ions and Fe recoils. The recoil spectrum is shown in Fig. A4.6 clearly distinguished recoils of H, N and Si and the film composition of H, N and Si were estimated as 3.8 at.%, 20.4 at.% and 75.7 at.% respectively.

![Figure A4.6: ERDA spectrum of a-SiN:H thin film using a stopper foil in tilted sample.](image)

**Impurities in Thin C Foil by Conventional ERDA**

The impurities in self supporting thin foil, used in nuclear physics experiments, were determined by ERDA in transmission geometry [39]. The recoil spectrum at 30° from a thin C foil for 10 MeV \textsuperscript{127}I ions, indicating the impurities (N, O, Na and Cl) is shown in Fig. A4.7. The scattered ions do not reach the detector placed at 30° as the maximum scattering angle for \textsuperscript{127}I on \textsuperscript{12}C is 5.4°.
Simultaneous Detection of Several Light Elements ERDA Using Telescope Detector

ERDA with conventional detectors provide the required information when the sample has elements with well separated masses. If the sample has elements with neighbouring masses, the recoil energies overlap and it becomes difficult to distinguish them. In such a situation, the particle identification techniques are utilized for the discrimination of different elements [40-42]. There are different possible configuration for telescope detectors. Transmission type thin solid state detector as $\Delta E$ detector and thick solid state detector as $E_{\text{rest}}$ detector is the simplest choice. But the solid state detectors are prone to radiation damage and thus have limited life in experiments. Second choice is to use a gaseous detector (which can be designed and fabricated according to the requirements) for $\Delta E$ along with a solid state detectors as the $E_{\text{rest}}$ detector. Third choice is to use the gaseous detector for both the $\Delta E$ and $E_{\text{rest}}$ detectors. Important feature and advantage of gaseous telescope detector is its being insensitive to radiation damage, rugged and can be fabricated indigenously. The telescope detectors are in use in major laboratories where SHIs are used for materials science such as LMU Munich [43], IUAC New Delhi [44], GSI Darmstadt [45], ANU

Figure A4.7: ERDA of a thin C foil using 100 MeV I ions, showing light mass impurities, which is possible only for self supporting thin film of a few tens of nm. [Reprinted from Jaipal, D. Kabiraj and D.K. Avasthi, *Nucl. Instr. and Meth.*, A 334 (1993) 196 with permission from Elsevier.]
Canberra [46] and Rossendorf Dresden [47]. Figure A4.8 is a two dimensional \(\Delta E-E\) ERDA spectrum [48] of a diamond-like carbon (DLC) film using such a telescope detector and 90 MeV Ni ions, which clearly shows the presence of N and O. The recoil energies from the surface of the sample for C, N and O recoils are well distinguished in the spectrum.

**Need of Large Area Position Sensitive Telescope Detector in ERDA**

Since there are possibilities of modification of sample in high energy heavy ion ERDA due to large \(S_e\), it is desirable to record the recoil spectrum with sufficient statistics using only a small fluence. To accomplish this objective, it is necessary to increase the solid angle of the telescope detector. However, the increase in the solid angle of the detector results in larger kinematic broadening, which in turn hampers the depth resolution. This problem is overcome by using a large area position sensitive detector and making kinematic correction by software utilizing the position of detected recoils [49]. The advantage of this approach is to increase the sensitivity without compromising the depth resolution. A schematic sketch of telescope detector is shown in Fig. A4.9. The
anode is split in two or three parts. The length of the anode is taken in such a way that the recoils of interest are stopped within this length when operating at a feasible gas pressure. Cathode is given a shape of backgammon so that the left and right side signals are created by the passage of recoil in the gas medium of detector to provide the information of position. The photograph of the on-line ERDA facility is shown in Fig. A4.10.

**Figure A4.9:** (a) Sectional view of the position sensitive ΔE-E telescope detector. The gas handling system is also shown. [Reprinted from D.K. Avasthi and W. Assmann, (Ed.) P. Chakroborty, Ion beam analysis of surfaces and interfaces of condensed matter systems. pp. 137, Nova Publisher.] (b) Schematic shows the anode, cathode Frisch grid which are main constituents of the telescope detector. All these are housed in a vacuum chamber shown in figure (a). [From Ref. (41) with permission from Indian Academy of Science.]

The anode is split in two or three parts. The length of the anode is taken in such a way that the recoils of interest are stopped within this length when operating at a feasible gas pressure. Cathode is given a shape of backgammon so that the left and right side signals are created by the passage of recoil in the gas medium of detector to provide the information of position. The photograph of the on-line ERDA facility is shown in Fig. A4.10.
On-line monitoring of ion induced modifications using a large area position sensitive detector [50], is one of the most interesting aspects of the ERDA.

**On-line Monitoring of Ion Induced Modifications**

On-line monitoring of ion induced modifications using a large area position sensitive detector [50], is one of the most interesting aspects of the ERDA. SHI is capable of producing modification at the surface, bulk and the interface.
of thin film and substrate. Electronic sputtering is an area which can be investigated by on-line monitoring in specific cases. The stoichiometric changes in the film, especially in the case of hydrogen and light element constituents, are also investigated by on-line ERDA. Modifying the sample by SHI irradiation and probing it at the same time is a unique feature of on-line ERDA.

**N Depletion Studies**

N loss in copper nitride film leads to the formation of nanoscale metallic structures in cupric nitride thin films by the impact of 200 MeV Au ions [51]. ERDA results show that the nitrogen content is reduced by 4.5 times due to irradiation to a fluence of $1.8 \times 10^{13}$ ions cm$^{-2}$. Conducting AFM showed the presence of conducting regions in the irradiated films, which could be due to production of copper-rich region as a result of the large nitrogen loss.

**Oxygen Content Measurements**

Majority of oxide materials studied by on-line ERD [52, 53] show that although there may be significant electronic sputtering but there is no preferential sputtering of oxygen. The studies show that 250 nm thick film of ZnO has sputter yield of 400 atoms/ion on irradiation with 100 MeV Au but the zinc to oxygen atomic ratio remains nearly constant throughout [54]. Fe$_2$O$_3$ [55], NiMn$_{0.05}$Ti$_{0.2}$MgFeO$_4$ [56], Li$_{0.25}$Mg$_{0.5}$Mn$_{0.1}$Fe$_{2.15}$O$_4$ [57], CuO [58] and nickel oxide [59] also do not show any preferential depletion of oxygen. Similar result is obtained in the case of GeO$_{1.7}$ thin films under 100 MeV Au ion irradiation [60]. Even though this system shows Ge phase separation due to SHI irradiation with the same beam [61]. However, sub-stoichiometric indium oxide film irradiated with 120 MeV Ag ions show preferential decrease of oxygen as well as phase separation leading to indium clusters of 35-45 nm size [62].

**Electronic Sputtering Measurements**

The large area gaseous telescope detector is used to measure the elemental content in the film at different fluences, which in turn gives the electronic sputtering [49, 50, 64-67] or desorption yield. A two dimensional ΔE-E spectrum is shown in Fig. A4.11 for the recoils from thin CaF$_2$ deposited on Si substrate, when 100 MeV Au ions are incident on the sample [63]. Integrated counts of Ca and F at different fluences are used to determine the electronic sputtering. At these high energies the contribution to sputtering due to nuclear energy loss is negligible and the sputtering in totally mediated by electronic energy loss. The desorption of carbon and hydrogen from amorphous C film was found to be dependent on the structural properties of the film. The electronic sputtering of LiF thin films [63, 68-70] were studied extensively by on-line ERDA. Typical spectra of LiF and CaF thin films on Si substrate are shown [63] in Fig. A4.11. The decrease in the number of recoils in individual elements with fluence allowed the measurement of electronic sputtering of thin halide films. A series
Figure A4.11: (a) A two dimensional ΔE-E spectrum for the recoils from BaF$_2$ thin film on Si substrate. (b) A two dimensional ΔE-E spectrum for the recoils from CaF$_2$ thin film on Si substrate. [Reprinted from Manvendra Kumar, Parasmani Rajput, S.A. Khan, D.K. Avasthi and A.C. Pandey, *Applied Surface Science*, 256 (2010) 2199 with permission from Elsevier.]
of experiments were performed on LiF, CaF$_2$ and BaF$_2$ thin films deposited on different substrates (glass, fused silica and Si) [63, 68-70]. Thickness of the films was 100 nm for all the films and the sputter yield of both the elements in the film was found to be stoichiometric. The total sputter yield, determined from ERDA areal concentration versus fluence curves, were $1.3 \times 10^5$, $2.5 \times 10^4$ and $1.2 \times 10^4$ atoms/ion, respectively from LiF, CaF$_2$ and BaF$_2$ films deposited on Si substrates, whereas they were $3.8 \times 10^5$, $7.8 \times 10^4$ and $3.7 \times 10^4$ atoms/ion respectively for LiF, CaF$_2$ and BaF$_2$ films deposited on glass substrates. No significant difference in the sputtering yield is observed for films on glass and fused silica substrates. The sputtering yield for film deposited on glass substrate is nearly three times higher than that on Si substrate for these halide thin films. The observed yields for different materials were compared as a function of band gap of the bulk materials. The band gap of bulk LiF, CaF$_2$ and BaF$_2$ are 14.2, 12.1 and 9.2 eV, respectively. It is clear that the yield increase as the band gap of the materials increases.

**On-line Monitoring of Mixing at Interface**

SHI produces mixing at the interface and the recoils provide information about the changes at the interface [49, 71]. For example, Cu recoil spectra indicated the mixing at the interface in an online ERDA measurement of CuO film on glass using 210 MeV I ion beam. The recoils detected in a large area position sensitive telescope detector and different masses appeared as different bands which were gated by software and the Cu recoil spectra at different fluences (in the beginning of irradiation and in the end of irradiation) was constructed, as shown in Fig. A4.12. The low energy region of the spectra represents the

![Recoil spectra of Fe (extracted from two dimensional ΔE-E spectra) obtained when 210 MeV I ion beam is incident on thin film of CuO deposited on glass.](image)

interface region. The change in the interface region is indication of the mixing caused by SHI’s at the interface. In another study [71], the mixing induced by 230 MeV Au ions incident on a thin Fe film deposited on a Si substrate was monitored on-line. With the application of kinematic correction and subsequent improvement in depth resolution, the detection system allowed the on-line study of ion beam mixing in Fe/Ti bilayer system using 135 MeV Au projectiles at room temperature. The decrease in the slope of the recoil spectra corresponding to Fe/Ti interface indicates mixing. For this system, the mixing rate was found to be $147 \pm 9 \text{ nm}^4$ [72].

**ERDA Channelling and Blocking Measurements**

Channelling ERDA experiments with energetic heavy ion beams are performed by detecting the recoils in forward direction. The experiment is carried out with the aligned sample to get the channelling scan by recording recoils (for a fixed incidence charge) at different angles. The channelling ERDA technique has been used to measure the strain [73] at the interface of CoSi$_2$ and Si crystal. When the recoils get blocked in crystallographic directions (by manipulating the sample using a goniometer), one records these recoils in the axial direction by a two dimensional position sensitive detector and a ‘shadow’ of the crystal axis appears. The shadow pattern is referred to as blocking pattern. Sample with high crystallinity gives rise to a blocking pattern with a very sharp contrast of the axes and the surrounding region. The blocking pattern for an amorphous sample will not show any axes. The contrast of the axis with the background gives the content of the crystallinity of the sample. ERD blocking has been used [74, 75] to investigate the radiation damage induced by swift heavy ions in semiconductor crystals.

**Energy Loss Measurements in ERDA Mode**

Energy loss ($dE/dx$) is a quantity of interest in characterization of sample by ion beam analysis such as RBS, ERDA, ion beam based cancer therapy, nuclear physics experiments like Doppler shift attenuation method (DSAM), etc. A large number of experiments [76-86] have been performed to measure the $dE/dx$ of light and heavy recoils upto Si in C and polymer films in different energy regimes. The recoils from a thin film act as ion beam for energy loss measurement in a foil of known thickness as shown in Fig. A4.13(a). A detector holding arrangement is shown in Fig. A4.13(b) where one detector is kept with the foil (in which $dE/dx$ is to be determined) and another without foil. Both the detectors make equal azimuthal angle from the plane of scattering [77]. The recoil spectra are recorded with and without foil using the detectors holding arrangement shown in Fig. A4.13(b). Other possibility is to use a detector covered half by the foil in which energy loss measurement is required.

The usefulness of RBS and RBS channelling is shown for determining the content of elements, sputtering, ion beam mixing and determination of strained layer superlattice. The use of detector telescope in ERDA allowed depth profiling of a wide range of elements up to mass 150 simultaneously. The large
Figure A4.13: (a) Schematic sketch of the energy loss measurements using the recoils. (b) Au arrangement to mount two detector. [Reproduction from A. Bhagwat and D.K. Avasthi, Jpn. J. Appl. Phys., 35 (1996) 313 with permission from Japan Society of Applied Physics.]
area position sensitive detectors with kinematics correction has been effective for keeping high sensitivity along with reasonable depth resolution, enabling the possibilities of on-line monitoring of the SHI induced changes at surface and at the interface of thin films on a substrate to probe electronic sputtering and SHI induced ion beam mixing. The experiment in ERDA mode using the recoils for energy loss measurement in thin foils/films have been effectively used.

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