

Particle Irradiation; An Innovative Tool in Materials Science

Bandyopadhyay SK

Retired Scientist, Variable Energy Cyclotron Centre, 6/2J, Naktala Road, Kolkata-700 047, India

ABSTRACT

Particle irradiation is the bombardment of materials with charged particles like alpha, heavy ion or neutrons. Primarily this creates various defects (like point defects like vacancy or line defects like dislocations) due to displacement of atoms. With the facility of charged particle irradiation at Variable Energy Cyclotron centre (VECC), we have carried out irradiation on variety of materials like Bi-based High Tc superconductors and have observed an increase in critical temperature (T_c) of the system. This was explained by particle irradiation induced oxygen knock out. There was also an increase in critical current density (J_c) which is having a great significance in context of application in devices.

In addition to charged particle irradiation on bulk materials, we have also undertaken heavy ion implantation on thin films of various materials like ZnO, and Ho, which have enhanced their magnetisation. Another dimension of ion implantation is irradiation induced growth of nanostructures like nanopillars. Irradiation induced nanopillars of Ni reflected enhancement of magnetization in the direction of growth of nanopillars.

*Corresponding author

Bandyopadhyay SK, Retired Scientist, Variable Energy Cyclotron Centre, 6/2J, Naktala Road, Kolkata-700 047, India.

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Introduction

With the advancement of science and technology, many innovative methods are being implemented for development of materials. No longer we adopt only simple chemical techniques governed by thermodynamic stabilities. Many exotic species are coming out as a result of these techniques [1-6]. Particle irradiation offers a new dimension of development and modification of materials to obtain desired properties [7-9]. It is the bombardment of materials with charged particles like alpha, heavy ion or neutrons. Primarily this creates various defects (like point defects like vacancy or line defects like dislocations) due to displacement of atoms [10-12].

There can be either penetration of particle through the target material if the kinetic energy is high or, medium (order of tens of MeV). In that case, the energy of particle is lost to create defects in target matrix. The defects modify properties of target material, often in the desired direction. The amount of energy lost depends on collision probability with target atoms, which depends on its energy or velocity.

If the energy of the projectile particle is low (few MeV or KeV), it gets stopped in the matrix of the target. This is implantation of projectile particle atoms in target material. Ion implantation is a regular practice employed in development of p and n type Si using Boron and Phosphorus ions respectively.

Irradiation Effect is quantified in terms of displacement per atom (dpa). $DPA = (\text{total no. of displacements}) / (\text{total no. of atoms})$. $\text{Total no. of displacements} = (\text{Total damage energy deposited}) / (\text{Displacement energy})$. Total damage energy can be assayed using simulation program Monte Carlo program SRIM (Simulation of Range of Ions in Matter) estimating differential cross section of projectile with target atoms as function of energy [13].

Consequences of Particle Irradiation are:

- Generation of point defects as vacancy or, interstitial atoms within the crystal lattice of the material being irradiated,
- Combination of vacancies to give rise to cluster of defects or void,
- Line defects in forms of various dislocations which play very significant roles in modification of material properties. This is elaborately seen in Figure 1.

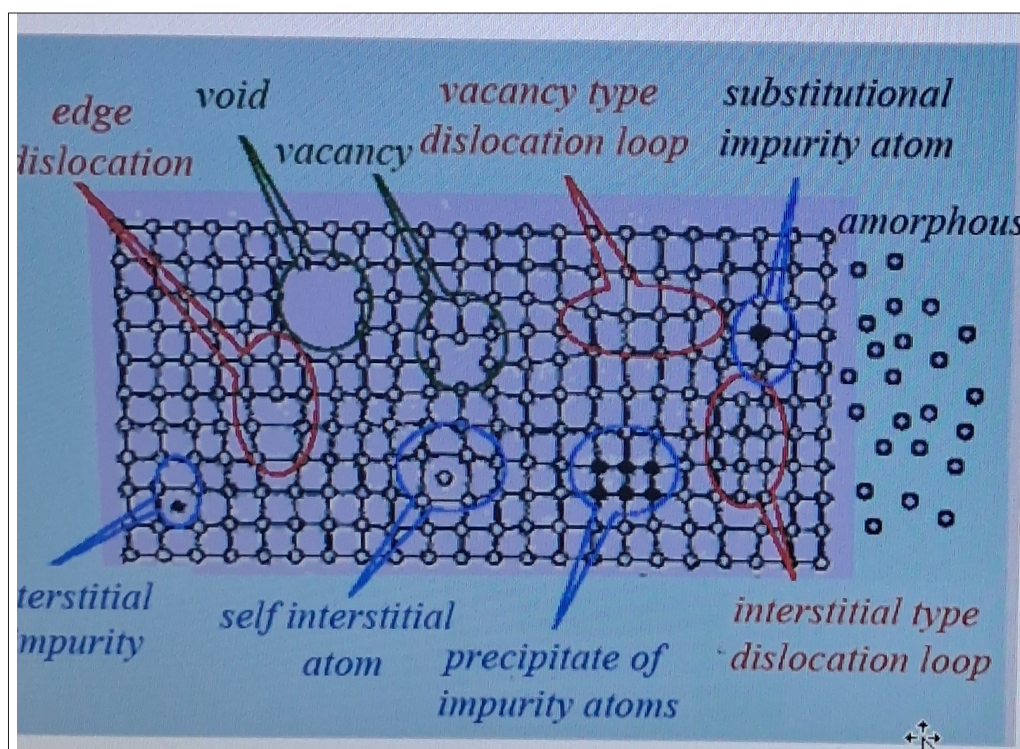


Figure 1: Consequences of Particle Irradiation with Medium Energy Particles

Another dimension opens up with irradiation induced growth, in particular with low energy ions. Formation of different types of nanostructures (nanoclusters) by ion irradiation & implantation. There can be periodic formation of surface structure of nanometer dimension by low energy ion irradiation. Sputtering of surface by low energy beam results in spontaneous pattern formation, giving rise to regularly spaced surface ripples and also different structures like nanopillars or nanorods.

In this paper, there is coverage of both medium energy and low energy irradiation/implantation. Section 1 covers the studies on medium energy particles (light ion as well as heavy ions) and Section 2 deals with low energy heavy ions.

Studies with Medium Energy Ions

We had observed an increase in critical temperature (T_c) in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$ (Bi-2212) high temperature superconductor (HTSC), which was so far unexpected, since particle irradiation induced defects generally cause a decrease in T_c [14]. This stimulated us to delve into search for increase in T_c in other superconductors as well as increase in critical current density (J_c) and their mechanisms. In this article, we are reviewing our activities of particle irradiation with different charged particles on various systems of superconductors. The results were quite encouraging in the context of application of these materials in devices.

Bi-2212

We had initially carried out irradiation of polycrystalline Bi-2212 with 40 MeV α -particles available at VECC. Range of 40 MeV α -particles in Bi-2212 is around 0.4mm as estimated from SRIM calculations [13]. To ensure uniformity, irradiation was carried out from both sides of the sample of thickness around 0.5 mm.

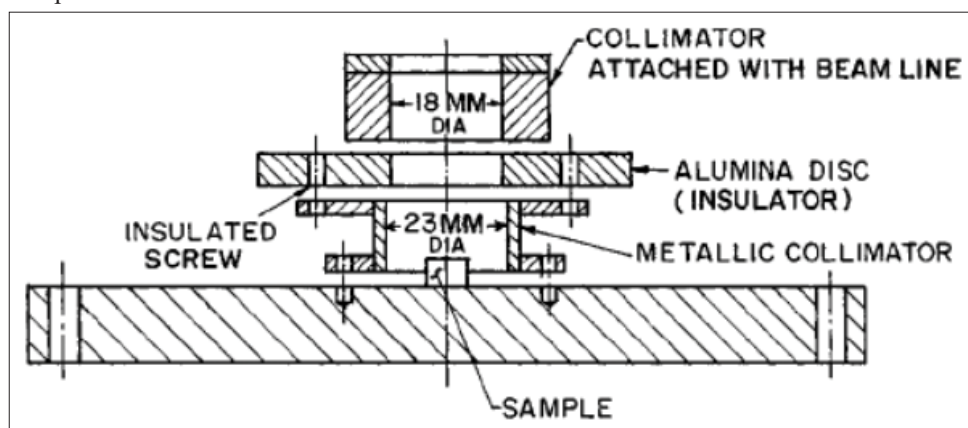


Figure 2: Target Assembly for Medium Energy Particle Irradiation with Cyclotron. Sample is Mounted on the Flange Insulated from the Body of Chamber and Charge Collected on the Flange is Measured and the Dose is Calculated from that

Figure 3 shows the Resistance versus temperature plots of the irradiated samples as a function of dose compared with the unirradiated one. At a dose of $\sim 10^{16} \alpha/\text{cm}^2$, the critical temperature T_c (i.e. the temperature where the resistance drops down to zero), increases from 65K of the unirradiated one to about 77K, quite appreciable. At further dose of irradiation, the material turned insulating.

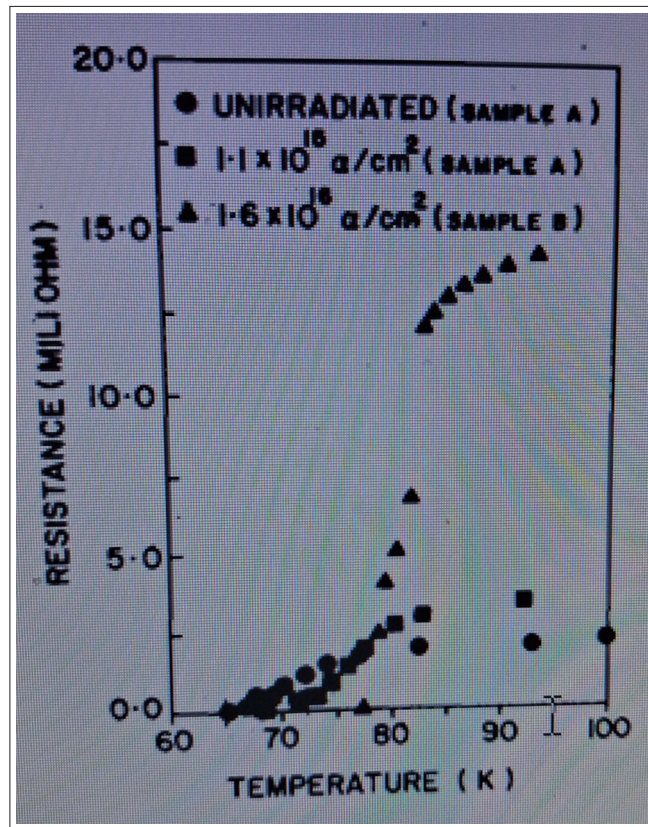


Figure 3: Resistance Versus Temperature for Unirradiated and Irradiated Bi-2212. Temperature Corresponding to Zero Resistance is Critical Temperature T_c

(Bi,Pb)-2223

We next studied the effect of 40 MeV α -particle irradiation on Bi-based High T_c superconductor (Bi,Pb)-2223 with higher T_c of 105K. But here there was no increase, rather decrease in T_c with particle irradiation as seen in Figure 4 [15]. In both cases, the oxygen content was decreasing with dose as measured by iodometry [16,17]. The difference of irradiation behaviour in these two systems was due to the role of excess oxygen in these two systems, which acts as carrier concentration. The crystal structures of these two systems are displayed in Figure 5. The structures containing so many layers are subjected to strain and the stability is governed by a tolerance factor given by

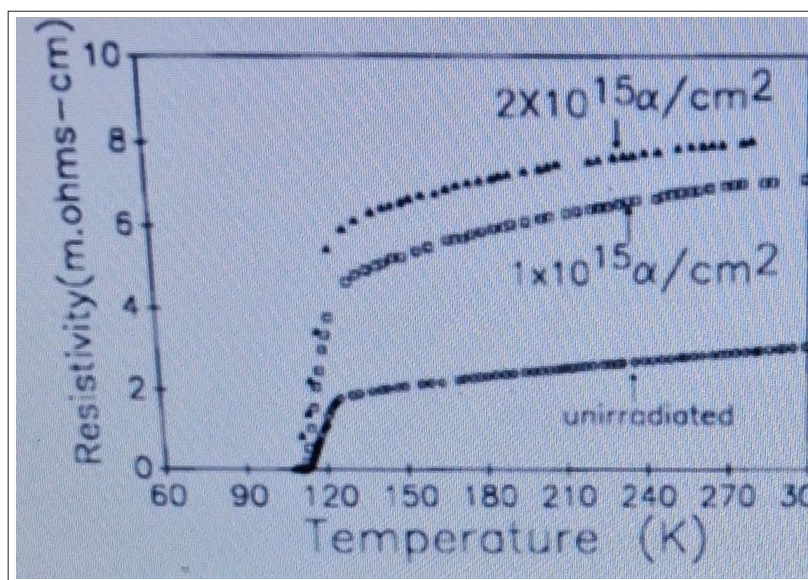


Figure 4: Resistivity Versus Temperature for Unirradiated as Well as Irradiated (Bi,Pb)-2223

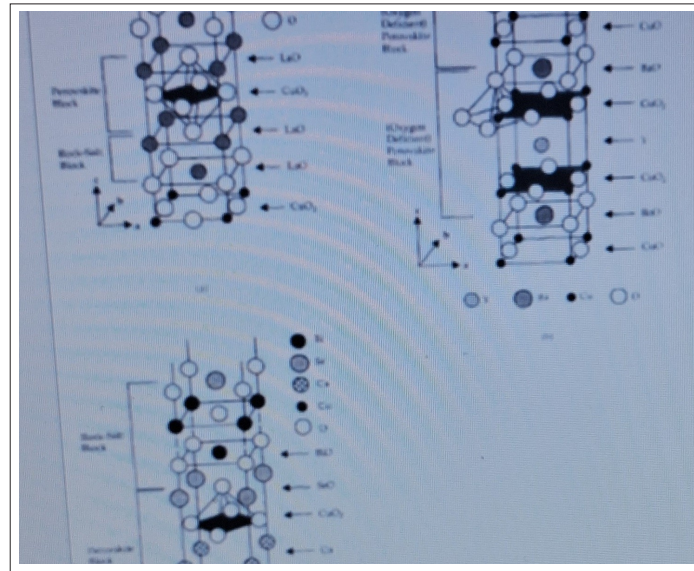


Figure 5: Crystal Structures of Bi-2212 and (Bi,Pb)-2223. Bi-O Layer Adjacent to Sr-O Layer

$$t = \frac{A - O}{\sqrt{2}(B - O)}$$

Bi-O layer adjacent to Cu-O layer of perovskite is A-O. Cu-O is B-O layer.

A-O bond distance is sum of ionic radii of Bi(III) and Oxide ion.

B-O is sum of Cu(II) and oxide ion.

Bi(III) being small, t is small, around 1.0

Cu-O bond is quite rigid and hence it cannot accommodate any extra oxygen. Bi-O accommodates extra O to relieve the structural strain at a distance of $0.7b$, the lattice parameter which gives rise to incommensurate modulation in crystal structure.

This extra oxygen is loosely bound and is vulnerable to be knocked out by particle irradiation.

In (Bi,Pb)-2223, Pb(II) larger ion partially substitutes Bi(III). Hence B-O is slightly larger and tolerance factor comes in the stability limit. Because of this structural stability gained by Pb(II) substitution, (Bi,Pb)-2223 is not vulnerable for irradiation induced oxygen knock out [16]. This excess oxygen in Bi-2212 plays the role of carrier concentration. The variation of T_c with this excess oxygen is shown in Figure 6. From the nature of variation of T_c with dose and consequently excess oxygen, it is clear that T_c increases with excess oxygen or carrier concentration and then decreases. This behaviour is noticed in other HTSC systems too like $YBa_2Cu_3O_{7-x}$.

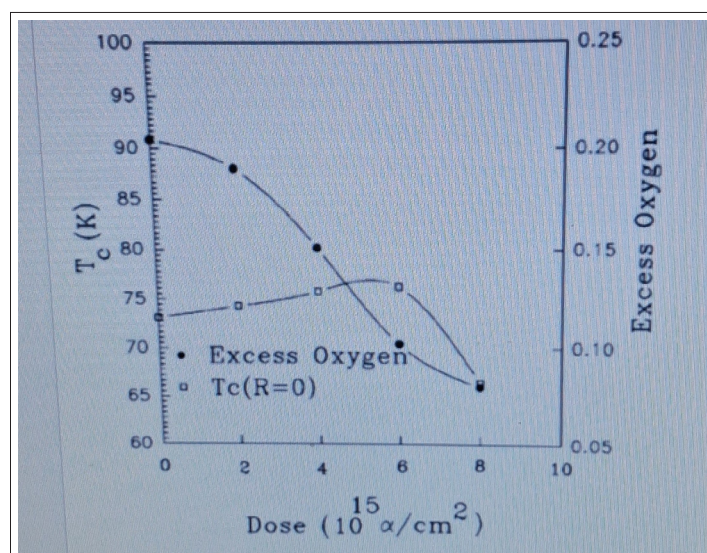


Figure 6: Variation of T_c and Excess Oxygen with dose of α -Particles in Bi-2212. The Excess Oxygen Decreases with Dose, but T_c Shows First Increase with Dose and then Decrease. The Excess Oxygen being the Source of Hole Carrier, it Reflects the Nature of Variation of T_c with Hole Carrier

Role of Irradiation Induced Oxygen Knock-Out

As noted earlier, the excess oxygen in these HTSC systems play the role of carrier and also the particle irradiation induced knock-out of oxygen plays the role of point defects in these systems causing a significant increase in critical current J_c due to pinning of magnetic flux in these type II superconductors. This can be assayed by measuring pinning potential, since J_c is closely related with pinning potential. Magnetoresistance measurements under magnetic field reveal the pinning potential at zero field. In figure 7, both magnetoresistance and the pinning potentials of 15 MeV proton irradiated Bi-2212 have been plotted. 15 MeV proton was employed considering its range larger than even 40 MeV alpha particles. Pinning potential has been estimated by Magnetoresistance measurement under different fields from the relation

$$\rho(T) = \rho_0 \exp[U(T,H)/kT]$$

$U(0,H)$ is obtained from the fit of $U(T,H)$ with temperature.

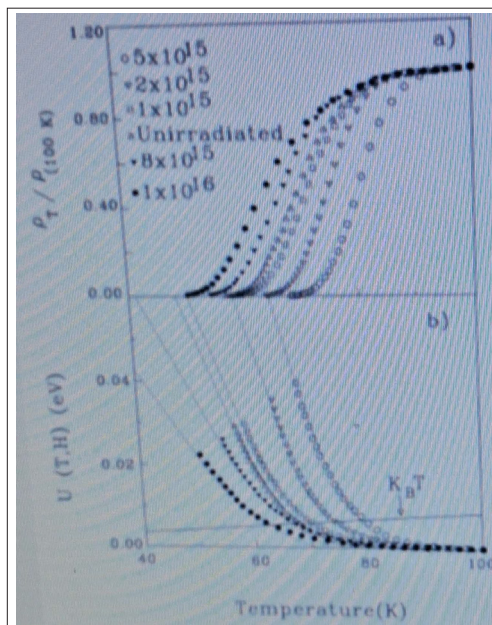


Figure 7: a) Magnetoresistance Versus Resistance Plot of Unirradiated as well as Proton Irradiated Bi-2212 as a Function of Dose

b) Corresponding Pinning Potentials Extracted from Magnetoresistance

We see that the pinning potential $U(0,H)$ increases with dose up to 5×10^{15} protons/cm² and then decreases. In case of (Bi, Pb)-2223, there is no significant increase with irradiation as revealed from Figure 8, where field dependence of activation energy $U(0, H)$ has been plotted against magnetic field as a function of dose for both Bi-2212 and (Bi, Pb)-2223 [18]. This is due to the availability of irradiation induced knocked out oxygen vacancies in Bi-2212 owing to the excess oxygen needed for its structural stability, whereas this is insignificant in (Bi,Pb)-2223 owing to its structural stability by partial substitution of Bi by larger Pb(II) ion [19]. This shows the significant roles of oxygen vacancies caused by particle irradiation in Bi-2212 in both increase of T_c and J_c . This is more relevant in Figure 9, where intragranular J_c as a function of dose has been plotted for Bi-2212 and (Bi, Pb)-2223. We notice a significant increase of J_c in Bi-2212 with irradiation up to 5×10^{15} protons/cm², but in (Bi,Pb)-2223, this is absent though the initial J_c of (Bi,Pb)-2223 is more [20]. This is due to high initial defect concn. in (Bi, Pb)-2223.

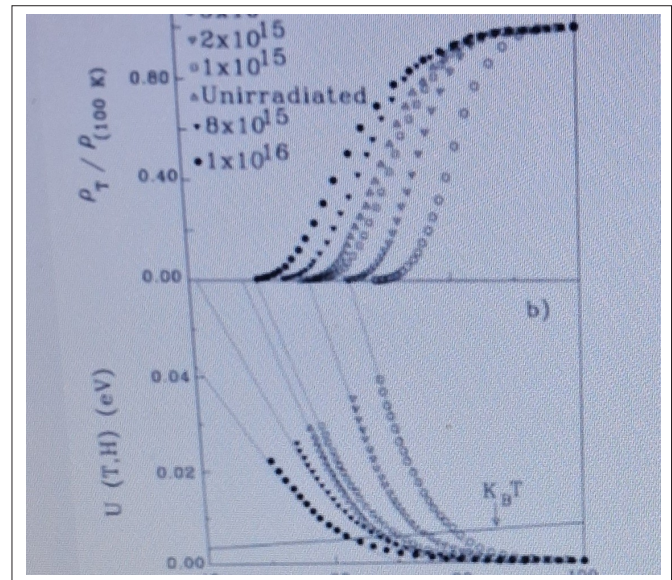


Figure 8: Field Dependence of the Activation Energy $U(0,H)$ in a) Bi-2212 and b) (Bi,Pb)-2223.

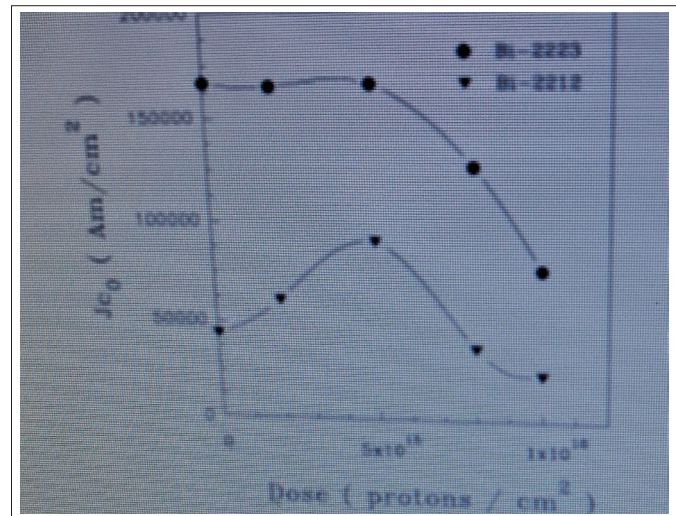


Figure 9: Intragranular J_c of Both Bi-2212 and (Bi,Pb)-2223 as a Function of Dose of Protons. The value of J_c is more for (Bi,Pb)-2223 but its increase with dose is not significant

Studies with Heavy Ions

In addition to light ions like alpha and proton, irradiation effects of heavy ions like oxygen on these Bi-based HTSC have been investigated [21]. Effects are similar in nature for both these systems.

In addition to Bi based HTSC, there have been particle irradiation studies on another superconductor MgB_2 , where the mechanism of superconductivity is not directly related with carrier concentration, but coupling between E_{2g} phonon with excess electron in B with sp^2 hybridised orbitals. Increase in J_c is there with heavy ion Ne irradiation as seen in Figure 10, but after certain field. Unlike the oxide based HTSC, there are no oxygen vacancies or defects. The non stoichiometric oxide based HTSC provides a lot of oxygen vacancies, enhanced by particle irradiation causing an increase in J_c and flux pinning potential.

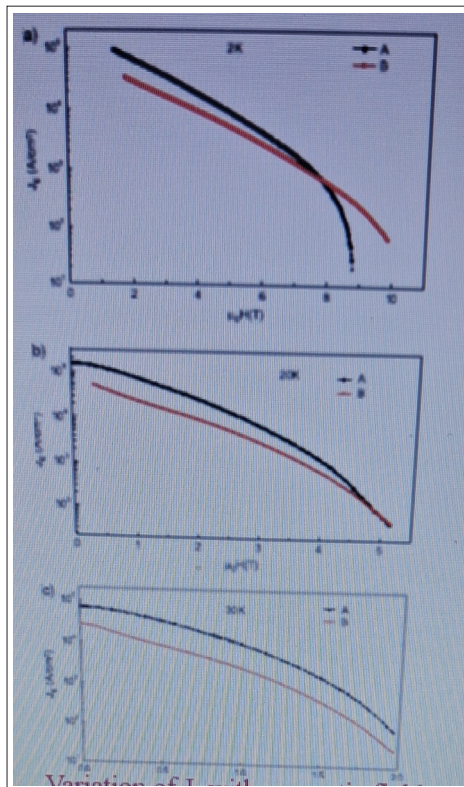


Figure 10: Variation of J_c with magnetic field at various temp for unirradiated (A) & Ne ion irradiated MgB_2 (B)

Irradiation/Implantation with Low Energy Heavy Ions

For low energy heavy ion irradiation/implantation, a separate beam line was used using ECR (Electron Cyclotron Resonance) based ion source was used with extraction voltage of around 10KV. From the ion source, the beam was passed through analysing magnet and quadrupole/dipole assemblies to obtain focussed beam. The beam line is displayed in Figure 11. Depending on the charge states of the ions, different energies of ions were obtained. Currents are optimized for individual charge states through a 20 mm wide image slit. For krypton and xenon ions the image slit width was decreased to 5 mm.

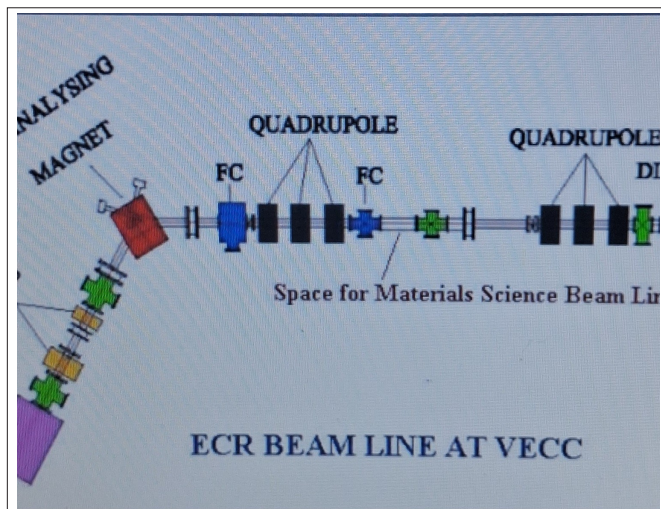


Figure 11: ECR Source Beam Line for Low Energy Ion Irradiation /Implantation. Ions of Variable Energies from Tens of keV to Hundreds of keV are Obtained

Currents are optimized for individual charge states through a 20 mm wide image slit. For krypton and xenon ions the image slit width was decreased to 5 mm.

Figure 12 shows the target assembly. Three targets can be mounted simultaneously. There is arrangement for the rotation of target assembly so that irradiation can be carried out at any desired angle.

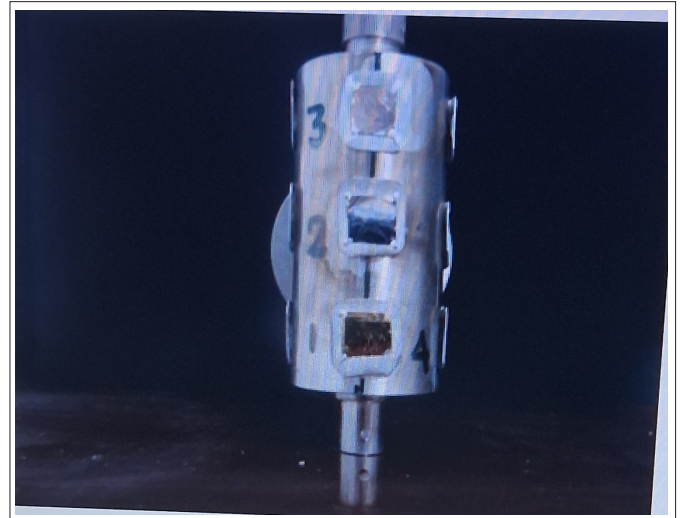


Figure 12: Target Assembly for Low Ion Irradiation. 3 Targets can be Mounted Simultaneously

Irradiation of Conducting Polymer with Ar Ion

Conducting polymers have manifold uses and often their dielectric constants are assayed along with real and imaginary impedances. Motivation of irradiation was to increase dielectric constant and dielectric loss, as these are of consideration for their application as Electromagnetic interference (EMI) shielding.

Polyaniline (PANI) prepared using water soluble support polymer polyvinyl alcohol (PVA) was subjected to irradiation with 150 keV Argon (Ar^{9+}) ion at the fluence of 7.68×10^{14} ion/cm² with beam current of 2 mA (electrical). There has been implantation of Ar atoms. The field dependence of dielectric response has been measured in the frequency range from 100 Hz to 1 MHz and in the temperature range from room temperature to the glass transition temperature [22]. Dielectric constants have been found to be more for the Ar^{9+} implanted polymer than for the pristine sample. Figure 13 shows the real and imaginary impedances of pristine PANI-PVA and Figure 14 the corresponding values for the Argon implanted PANI-PVA. It is observed that dielectric constant and ϵ'' (eq. to loss) increases after Ar Ion implantation [23]. This is significant in application for EMI (Electromagnetic induction) shielding in devices [24].

The frequency dependence of the imaginary complex impedance is found to obey Arrhenius law with an activation energy of 0.29 eV for unirradiated and 0.53 eV for the Ar^{9+} implanted polymer. Using the Cole-Cole model, an analysis of the real and imaginary parts of the impedance is performed, which reflects an increase in poly dispersive nature after irradiation.

Resistivity analysis shows three-dimensional (3D) Variable Range Hopping (VRH) at low temperature in contrast with two-dimensional (2D) VRH. There has been a decrease in the localization length as well as an increase in the energy gap due to the scission of bonds by the energy deposited by Ar^{9+} ions.

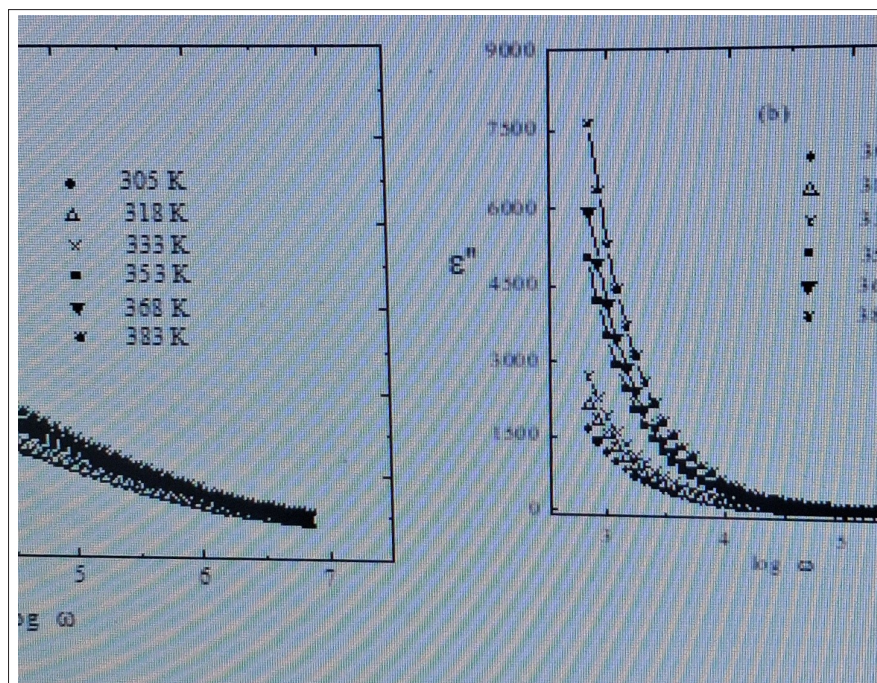


Figure 13: Variation of Real & Imaginary Parts of Dielectric Constant for Pristine PANI-PVA with Frequency at Different Temperatures.

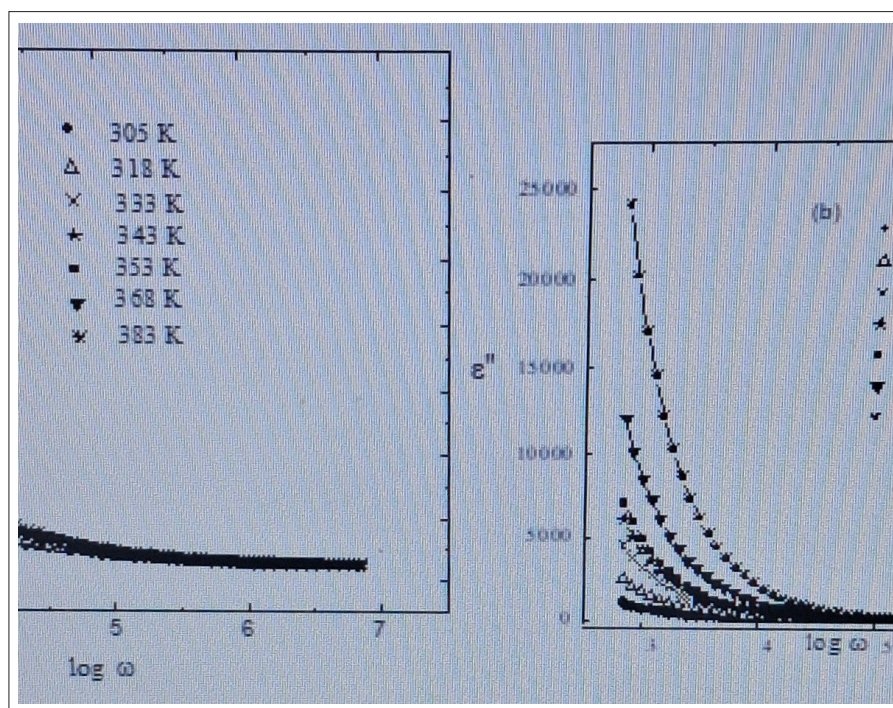


Figure 14: Variation of Real & Imaginary Parts of Dielectric Constant for Ar Implanted PANI-PVA with Frequency at Different Temperatures

The frequency dependence of the imaginary complex impedance is found to obey Arrhenius law with an activation energy of 0.29 eV for unirradiated and 0.53 eV for the Ar^{9+} implanted polymer. Using the Cole–Cole model, an analysis of the real and imaginary parts of the impedance is performed, which reflects an increase in poly dispersive nature after irradiation.

Resistivity analysis shows three-dimensional (3D) Variable Range Hopping (VRH) at low temperature in contrast with two-dimensional (2D) VRH. There has been a decrease in the localization length as well as an increase in the energy gap due to the scission of bonds by the energy deposited by Ar^{9+} ions.

Irradiation of ZnO Films

Motivation was to induce magnetisation in ZnO films by irradiation induced oxygen vacancies. Thin films of ZnO were grown by RF sputtering technique on Aluminium foil at VECC using polycrystalline ZnO annealed at 900°C. Thickness of the films were in the range 80-90nm as measured by energy loss of α -particles. 150 keV Ar⁹⁺ ions from ECR source at VEC were used having a range of 90nm in ZnO as obtained from SRIM calculations [13]. The oxygen vacancies generated by 150keV Ar ions is quite high. Beam current employed was 2 μ A (electrical). Irradiation was carried out at various fluences from 10¹³ to 10¹⁶ ions/cm². Magnetisation was studied by M-T measurements from room temperature to 5K by zero field cooling and field cooling at 100 Oe using Quantum Design SQUID Magnetometer. There was no sign of magnetisation up to the fluence of 1x10¹⁵ ions/cm². At the fluence of 1x10¹⁶ ions/cm², there was a signature of magnetisation in field cooled curve of M-T studies as shown in Figure 15.

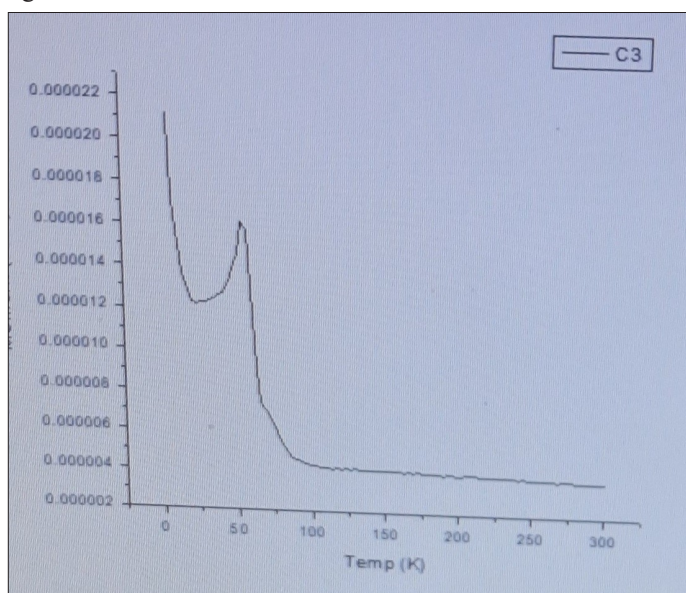


Figure 15: M-T Curve (FC) for ZnO Film Irradiated at 1x10¹⁶ ions/cm²

Ion Irradiation to Grow Nanostructures

Low energy ions can be effective for the formation of different structures like ripples, nanopillars etc. For this, glancing angle irradiation is effective. There can be periodic formation of surface structure of nanometer dimension by low energy ion irradiation.

Sputtering of surface by low energy beam in glancing angle results in spontaneous pattern formation, giving rise to regularly spaced surface ripples [25].

We took Ni thin foils with a view to having an effect on magnetisation induced by the growth of nanostructure. Argon of 8keV energy was employed. Since glancing angle irradiation is better for development of nanostructure, we carried out irradiation at different angles of incident beam. There was development of nanostructure in the form of nanopillars, particularly when the fluence was increased to 7x10¹⁶ ions/cm². The structures are shown in Figure 16 and Figure 17 for the fluence of 5x10¹⁶ and 7x10¹⁶ ions/cm² respectively. The growth of pillar was in the c-direction, the natural axis of magnetisation. We observed an increase of magnetisation after irradiation.

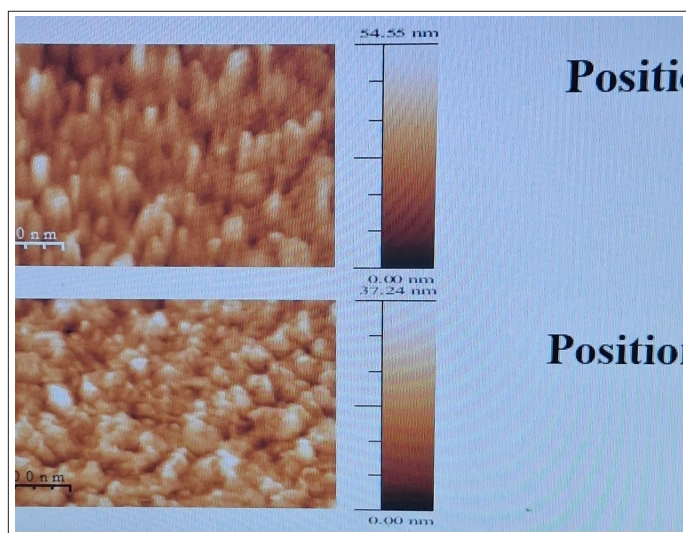


Figure 16: Nanostructure of Ni Bombarded at 100 with 8keV Ar⁺ with Fluence of 5x10¹⁶ ions/cm²

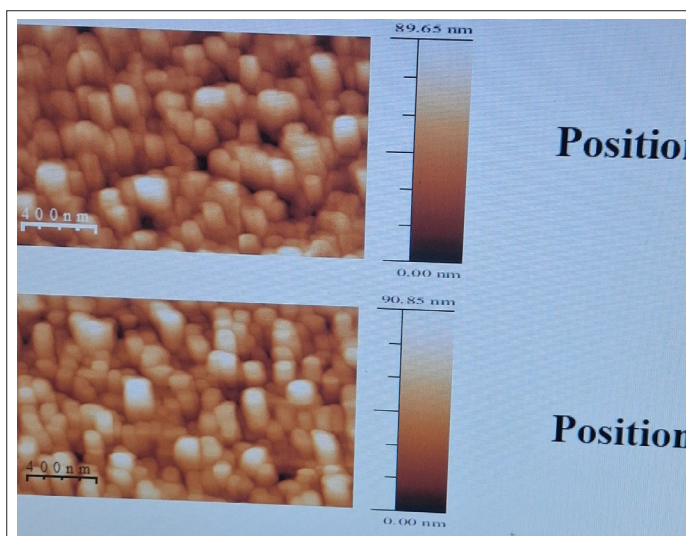


Figure 17: Nanostructure of Ni Bombarded at 100 with 8keV Ar⁺ with fluence of 7x10¹⁶ ions/cm²

Conclusions

We have undertaken charged particle irradiation at VECC with both medium energy 40 MeV α and 15 MeV protons and low energy ions from ECR ion source available at VECC. The medium energy particles were employed for irradiation of bulk polycrystalline Bi-2212 and (Bi-Pb)-2223 samples. A remarkable difference with respect to particle irradiation was noticed in these two systems. In case of Bi-2212, irradiation induced knock-out of oxygen was significant, whereas it was quite insignificant in case of (Bi, Pb)-2223. It is because of the accommodation of extra loose oxygen at Bi-O layer in Bi-2212 to relieve structural strain. This oxygen is loosely bound and gets knocked out by particle irradiation giving rise to oxygen vacancies which plays a crucial role in controlling T_c and J_c. On the other hand, in (Bi, Pb)-2223, the structural stability is achieved by partial substitution of Bi(III) by larger Pb(II) ion and it does not need extra oxygen and hence it does not play significant role in creating oxygen vacancies by particle irradiation. Hence there is no appreciable change in J_c in (Bi, Pb)-2223, though the pristine material is having higher J_c owing to structural defects. In case of MgB₂ superconductor, the mechanism of superconductivity is different from that of Bi-based

HTSC, where excess oxygen plays the role of hole carrier. In MgB₂ with hexagonal structure, there is coupling between E_{2g} phonon and electron of B with electrons from Mg.

We have carried out low energy ion irradiation mostly with Ar ions at different energies. Conducting polymer based on PANI-PVA was subjected to irradiation with 150keV Ar⁹⁺ ions to investigate the changes in dielectric properties. Analysis of real and imaginary impedances revealed that dielectric constant and ϵ'' (eq. to loss) increases after Ar Ion implantation which is quite important in EMI shielding. Ar ion irradiation on thin films of ZnO brought in magnetisation at a fluence of the order of 10¹⁶ ions/cm². We also attempted to develop nanostructure in Ni film. There were developments of nanopillar like structure with ion beam kept at a glancing angle of 100 of the incident beam with target. This nanopillar displayed enhanced magnetisation along c-axis of Ni film. These were the manifestations of ion irradiation in various dimensions which are indications of the application of particle irradiation in various devices.

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