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Toward Single-Phase Bi-2223: Integrating Ion Irradiation with Advanced Processing of Cuprate Superconductors

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Abstract: *Attaining high T_cBi-2223 phase in Bi-Pb-Sr-Ca-Cu-O (BPSCCO) thin films is complicated. The system tries to form several phases and grain boundaries, thus limiting film quality. Furthermore, Bi-2223 phase is innately metastable, making it difficult to stabilize throughout growth. This study gives an in-depth and up-to-date overview of the most recent methods for fabricating and post processing BPSCCO films, with an emphasis on approaches that increase the formation, stability and overall quality of the high T_c phase in thin films. We present ion irradiation as a promising method for controlling phase development in BPSCCO films. Different irradiation techniques are examined for their ability to reorganize the microstructure, drive phase change and suppress lower -Tc phases. The simultaneous use of ion irradiation and other processing methods such as chemical doping, thermal annealing and laser treatment is thoroughly investigated. Emphasis is given on how these hybrid strategies can enhance phase selectivity and improve superconducting performance. Although the reports of direct conversion of mixed- phase films to single phase Bi-2223 using irradiation is rare, multistep approaches continue to yield promising outcomes. Major challenges in optimizing irradiation parameters and understanding the underlying mechanisms and how these gaps will require real time characterization and well- structured experimental studies, are discussed. The review concludes by outlining future research directions that could enable the controlled and reliable synthesis of high quality Bi-2223 films for next generation high- Tc super conducting technologies.*

Keywords: *BPSCCO thin films, Bi ⁻2223 phase, High-Tc superconductivity, Ion irradiation, Phase engineering, Chemical doping, Thermal annealing, Oxygen implantation, Defect-assisted diffusion, Mixed-phase superconductors*

I. Introduction

High- temperature superconductors(HTS) continue to draw strong interest in physics and material science as they can carry electricity with zero resistance at relatively high critical temperatures (Tc). The bismuth based cuprates (BSCCO: Bi–Sr–Ca–Cu–O) are especially important. They are easier to fabricate than many other superconductors, absence of rare earth elements, and their strong anisotropy makes them useful for some specific designs [1]. The BSCCO family contains several distinct phases viz $Bi_2Sr_2CuO_{6+\delta}$ (Bi-2201, Tc ≈ 30 K) $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212, Tc ≈ 85 K) and $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (Bi-2223, Tc ≈ 110 K). Of these, Bi-2223 is particularly appealing for technological applications. Its higher Tc enables efficient operation near liquid nitrogen temperatures, reducing cooling costs and broadening real-world applications [2].

Even though Bi-2223 is a promising high -T_c material, synthesis of single phase 2223 thin films is extremely difficult. Common deposition methods such as pulsed laser deposition, sputtering and chemical solution techniques generally produce mixed Bi-2212/Bi-2223 compositions. As a result of this the superconducting transition temperature (ΔTc) becomes broader and the critical current density (Jc) drops. Overall the device performance is compromised due to interphase boundaries and weak links between grains [4]. These ongoing challenges highlight the need to explore alternative processes that can stabilize the Bi-2223 phase and also improve the microstructure of the films.

This review takes up the challenge by examining the ion irradiation as a post deposition method for improving BSCCO thin films. Although irradiation was initially employed to create defects and enhancing flux pinning, recent studies show that it may also help to stabilize phases and improve the microstructure. By reviewing its mechanism, reported success and integration with chemical doping and heat treatment, the article discusses where ion irradiation fits into broader phase research initiatives. The discussion emphasizes the possible benefits and the obstacles, offering important information for researchers working towards reliable methods to make high Tc Bi-2223 films with improved performance.

Challenges in Bi-2223 Phase Stabilization

The primary reason to synthesize single-phase Bi-2223 is that Bi-2212 and Bi-2223 have a very small free-energy difference and almost the same lattice structure [5]. As a result of this, Bi-2212 tends to form more easily during film growth. Process of formation of Bi-2223 is also slow and requires long annealing times under very precise temperature and oxygen conditions. Problems at grain boundaries worsen the situation: misaligned grains act like weak links lowering the critical current density, Jc [6]. Moreover, oxygen is distributed unevenly and Bi, Ca, and Sr may evaporate or migrate during heating which destabilize Bi-2223. It results in breakdown of Bi-2223 into Bi-2212 or undesired phases such as $(Sr,Ca)CuO_2$ and CuO [7].

Various methods of film-growth have been tested to overcome these challenges. In Pulsed laser deposition (PLD) exact composition from ceramic target is transferred. But, as the plasma plume cools quickly and sometimes due to the substrate mismatch, it tends to form Bi-2212 [8]. RF/DC sputtering requires precise oxygen control to avoid undesirable phases [9]. Chemical methods like metal-organic deposition (MOD) and sol-gel offer good control over composition. Adding dopants like Pb or Sb helps stabilize the Bi-2223 phase [10]. Other methods, which includes spray pyrolysis [11] and chemical vapour deposition (CVD) [12], can coat large areas, but achieving high phase purity remains difficult. Across all these techniques, achieving stable Bi-2223 still depends on combining the deposition step with very careful post-processing. Each method has its strengths. Chemical techniques are low cost, physical methods give better texture and hybrid approaches improve composition. None of these methods consistently produce single-phase Bi-2223 on their own. This suggests that additional post-treatments, such as ion irradiation, are needed to further refine the phase and improve film quality.

Technique	Key Advantages	Limitations for Bi-2223 Phase
Pulsed Laser Deposition	Stoichiometric transfer, high control	Favours Bi-2223 due to plume quenching, substrate strain
RF/DC Sputtering	Scalable, uniform films	Requires stringent oxygen control
MOD / Sol–Gel	Flexible chemistry, Pb/Sbdoping enhances Bi-2223	Strong sensitivity to annealing atmosphere
Spray Pyrolysis	Continuous coating, adaptable	Limited phase selectivity
Chemical Vapor Deposition (CVD)	Large-area growth	Phase purity difficult; parasitic phases likely

Table 1 Deposition techniques and their influence on Bi-2223 stabilization

Strategies to Enhance High-Tc Phase in Mixed BSCCO Films

Achieving a pure single- phase Bi-2223 film is still very difficult, but several approaches like chemical doping, phase control and careful annealing have helped in increasing the amount of Bi-2223 and improving superconducting properties. Among these, adding Pb at the Bi site has been the most effective. Pb substitution reduces the energy needed to form Bi-2223. It improves the diffusion of Ca and Cu atoms, and helps suppress unwanted phases with the optimal results for $x \approx 0.1$-0.3 in $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ [13]. Adding small amounts of Sb further improves grain boundaries and reduces the loss of Bi during processing. Films co-doped with both Pb and Sb show sharper superconducting transitions and higher Jc [14]. Rare-earth elements such as Yb, Sm, and Pr have also been used to adjust lattice strain and oxygen movement, e.g. Yb^{3+} substitution shifts the balance toward Bi-2223 and improves grain alignment, especially when combined with Pb [15].

Post-deposition annealing is another step for stabilizing the Bi-2223 phase. High-temperature annealing in oxygen (500-850 ^oC) helps the oxygen uptake, improves cation diffusion and supports the growth of Bi-2223. However, the results depend strongly on factors such as heating rate, duration, oxygen pressure and even the choice of substrate. Long anneals with slow temperature ramps (20-100 h) and high oxygen pressure (>1 atm) work best. Substrates like $LaAlO_3$ can help promote oriented grain growth. Complete conversion to Bi-2223 usually requires Pb doping [16]. Beyond standard annealing, excimer-laser annealing (KrF, 248nm) can selectively modify only the top layer of the film by rapid localized heating. It reduces the weak links between grains, makes the superconducting transition ΔT_csharper and increases the amount of Bi-2223 near the surface. When used together with Pb/Sb doping, laser annealing greatly improves the texture and J_c [17]. Overall, chemical doping, thermal annealing and laser treatment work together to improve phase purity, microstructure and superconducting behaviour.These improved starting point makes later treatments such as ion irradiation much more effective in taking the film further towards Bi-2223 phase.

Ion Irradiation in BSCCO Films: Mechanisms and Effects

Ion irradiation has emerged as a robust post-deposition tool to tune the Bi-2212/Bi-2223 phase balance in BSCCO films and enhance superconducting performance. By introducing controlled defects, enhancing oxygen diffusion, and locally restructuring the lattice, irradiation can destabilize Bi-2212 and promote Bi-2223 nucleation. Its effectiveness is strongly governed by ion species, fluence, energy, and irradiation temperature.

Ions such as O^+, Ar^+, He^+, Au^+, or Xe^+ hit the film with high energy creating collision cascades and small regions of local heating. Defects, vacancies, interstitials and strain in the lattice [18] are created causing easy movement of oxygen and metal atom. It helps in the growth of Bi-2223 phase. Irradiating at higher temperatures (200-300 ^oC) helps in defect migration and oxygen redistribution which stabilizes the Bi-2223 phase [19]. In contrast, light ions (H^+, D^+) causes excitation of electrons and loss of oxygen which leads to degradation of superconductivity. Deuterium irradiation has been shown to reduce J_c by 50% and broadened T_c

transitions [20]. Medium-energy protons (MeV range) create well-ordered defect patterns that act as strong flux-pinning sites. These defects can significantly increase Jc while reducing T_c by less than 2K at fluences of approx.10^{16} cm^{-2} [22]. Oxygen ions behave differently from other ions as they can directly modify the lattice stoichiometry. When oxygen is implanted at 300-600 ^oC, redistribution of oxygen inside it takes place and the film remains crystalline. This shift the Tc by 10-15 K and support the formation of the Bi- 2223 phase, although full conversion usually needs additional doping or annealing steps [23]

Fluence of ions also plays a major role. Doses below $10^{11}cm^{-2}$ yields no effect, while above 10^{14} cm^{-2} can damage the film and even cause amorphization. The most optimal range lies between 10^{12}–10^{13} cm^{-2}. Ion Energy also matters as it defines penetration depth. low energies (<1 MeV) affect surfaces, medium energy (10–50 MeV) modifies sublayers, and high energy (100–200 MeV) causes uniform modification.

The light ion irradiation is not suitable for improving the phase quality of BSCCO. Instead, it mainly helps researchers understand how radiation damages the material, how superconductivity degrades under particle exposure, and how oxygen moves inside the lattice. But for strengthening or stabilizing the high Tc Bi-2223 phase, light ions such as H^+ and D^+ are counterproductive and usually reduce performance. Therefore, if the goal is for functional enhancement, it is better to use heavier ions that create stronger lattice interactions. These ions should be used at moderate fluence levels and ideally applied after chemical doping and thermal pre-treatments, which help the material respond more effectively to irradiation.

Despite progress, ion irradiation by itself has rarely produced a completely single-phase Bi-2223 film. Its main strengths are in creating defects, improving the microstructure and increasing flux pinning. A rare study did report a Tc increase of about 15 K in BPSCCO films after high fluenceAr-ion irradiation [21]. However, most researchers agree that irradiation works best when combined with other treatments such as Pb/Sb doping, textured substrates and controlled annealing.

Ion species	**Energy range**	**Optimal fluence**	**Key effects**	**Impact on Tc / Jc / Phase**
H^+, D^+ (light)	0.1–1 MeV	~10^{13} cm^{-2}	Electronic excitation, oxygen loss	Tc ↓, Jc ↓, Bi-2223 suppressed
Protons (MeV)	1–5 MeV	~10^{16} cm^{-2}	Defect arrays, flux pinning	Tc stable (–2 K), Jc ↑↑
Au^+, Xe^+ (heavy)	100–200 MeV	10^{12}–10^{13} cm^{-2}	Columnar tracks, strain, grain texturing	Tc stable, Jc ↑, Bi-2223 fraction ↑
O^+ implantation	100–200 keV	10^{16}–10^{17} cm^{-2}	Oxygen redistribution, stoichiometry tuning	Tc shift (+10–15 K), Bi-2223 promoted (partial)
Ar^+ (case study)	100 MeV	High fluence (>10^{15} cm^{-2})	Localized lattice annealing	Tc ↑ (~15 K), near single-phase Bi-2223 reported

Table 1. Summary of ion irradiation effects in BSCCO films

Comparative Analysis: Ion Irradiation vs Other Techniques for High-T_c Bi-2223 Films

To obtain phase–pure Bi-2223 films, chemical stability, microstructural quality, and oxygen stoichiometry, must all be balanced out. Conventional techniques, such as Pb-based chemical doping, thermal annealing and advances deposition, have all contributed, but they also have limitations.

Technique	**Primary Role**	**Advantages**	**Limitations**	**Typical Outcome for Bi-2223**
Pb (±Sb/Re) Doping [24]	Thermodynamic stabilization of Bi-2223	Raises Tc up to ~110 K; promotes grain growth; tunes carrier concentration	Requires strict stoichiometry; risk of secondary phases; does not fix grain boundary misalignment	Improved Tc, partial suppression of Bi-2212
Thermal Annealing (O_2, Furnace) [25]	Phase crystallization&oxygen optimization	Improves phase formation and connectivity; enhances oxygen stoichiometry	Long high-T cycles → secondary phases, inter diffusion, grain coarsening	Bi-2223 growth promoted but incomplete; weak pinning persists
Laser Annealing	Localized recrystallization	Fast, spatially selective; reduces weak links	Limited penetration depth; requires precise tuning	Surface phase uniformity improved
Deposition (PLD, MOCVD, Sputtering) [26]	Film formation&phase control	Precise thickness/composition control; scalable; compatible with biaxially textured substrates	Sensitive to parameters (T, pO_2); lattice mismatch, cracks, mixed phases common	Oriented films with partial Bi-2223; often need post-annealing

Ion Irradiation (H^+, Au^+, Xe^+) [27]	Defect/oxygen redistribution; flux pinning	Enhances Jc under magnetic fields; grain realignment; can suppress Bi-2212 when combined with annealing	Risk of amorphization; Tc reduction if over-irradiated; rarely achieves single-phase alone	Enhanced pinning, partial Bi-2223 stabilization
Oxygen Implantation	Local stoichiometry tuning	Directly modifies oxygen content; enables patterned Tc control	Requires high-T to avoid amorphization; incomplete phase conversion alone	Tc tuning, localized modification, potential Bi-2223 nucleation

Ion Irradiation: Unique Role and Limitations

Ion irradiation is one of the efficient and impactful method to bring-out change in the microstructure and electronic properties of BPSCCO films. One of its most characteristic features is its potential to produce controlled nanoscaledefects. It functions as efficient flux pinning sites that enhances the critical current density (J_c). Ion beams can also change grain boundary characteristics and promote preferred grain orientation through processes like ion-beam structure modification (ISM). It has shown promise when applied to bi-axially textured metal substrates. In addition to microstructural control, ion irradiation can also enable localized tuning of phase composition, facilitate oxygen redistribution, and allow nanoscale patterning of superconducting regions when paired with techniques like oxygen ion implantation.

II. Results and Discussion

To investigate how ion irradiation affects the critical current density (J_c) and superconducting transition temperature (T_c) of BSCCO based films, typical data was obtained from published experimental research on proton, oxygen and heavy-ion irradiation in Bi-2212/Bi-2223 systems. These datasets were drawn from peer-reviewed studies on irradiated BSCCO films, tapes and bulk materials and utilized to create the comparison plots. However, many studies report only relative changes rather than exact numerical values, limiting the precision and completeness of dataset.

1. Effect of irradiation on critical current density J_c

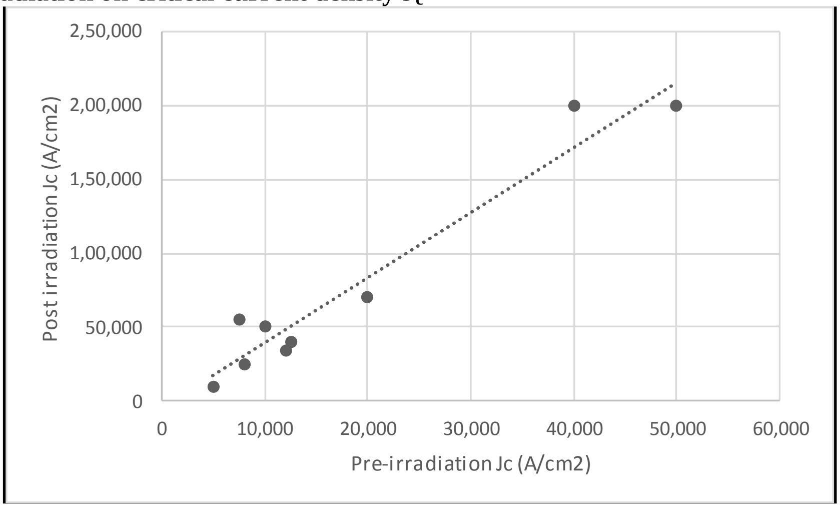

Fig 1: Critical current density J_c, before and after irradiation

The scatter plot (Fig.1) shows the effect of irradiation on critical current (J_c) of the materials. Each point on the plot represents a single material sample. The dashed line shows best fit line regression. A positive correlation can be observed from the graph. Improvement is seen in samples which have higher J_c before irradiation. This suggests improvement in microstructural quality due to ion irradiation rather than formation of new pathways. Effective pinning centres are introduced in well-connected films with considerable Bi-2223 content after irradiation which raised J_c substantially, pushing the data above regression line. Samples which had poor connectivity show negligible improvement or slight degradation. This shows that excessive disorder or insufficient recovery annealing prevail over any gains. In general, the plot shows that irradiation functions as a performance enhancer, particularly when applied to films with adequate phase purity and microstructural integrity.

2. Effect of irradiation on critical Temperature T_c

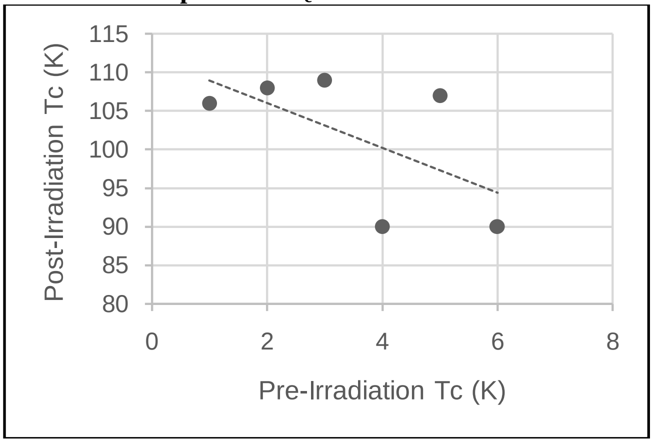

Fig 2: Critical Temperature T_c before and after irradiation

The plot shows the influence of irradiation on critical temperature T_c of the material samples (Fig.2). Each point on the graph represents a distinct experiment reported in literature. The dashed line indicates the overall tendency. The data points show that T_c is largely preserved by irradiation. Bi-2223 rich samples which have T_c = 110K initially, show relatively minor variations of about ± 1K. The graph also shows that one Bi-2212 rich sample which has an initial T_c of 92 K experiences a significant decrease in T_c after irradiation. This implies that certain irradiation conditions, particularly with light ions or high fluence, can introduce disorder and destabilize oxygen content which suppresses the superconductivity. In general, the trend suggests that under optimal conditions, irradiation can maintain or slightly improve T_c. It can also reduce Tc when the material or irradiation parameters are not ideal.

3. Effect of fluence on critical current density J_c ratio

The influence of ion irradiation on superconducting transport can be successfully evaluated by comparing the change in critical current density J_c as a function of irradiationfluence. The scatterplot (Fig 3) illustrates the correlation between the change in critical current density $J_c$and the irradiation fluence (ratio of post-irradiation J_c to pre irradiation J_c). The scatter in the plot reflects the diverse irradiation conditions such as ion types, energies, sample composition and processing history, used across studies.

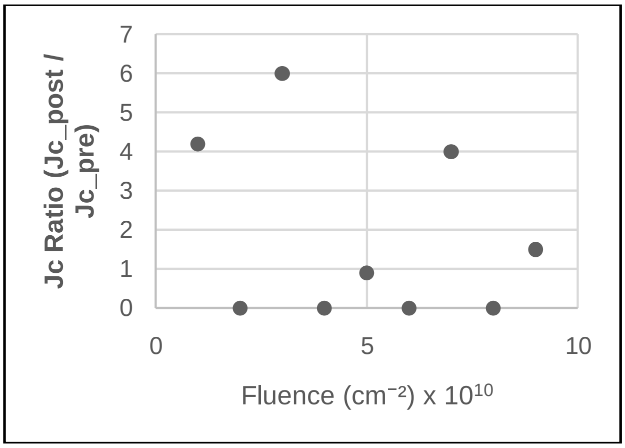

Fig 3: Variation of fluence with critical current density $J_c$ratio

The plot shows that there is a general trend of increasing J_c ratio with higher fluence, suggesting that a larger dose of irradiation can lead to a greater improvement in the critical current density. The graph indicates that the maximum enhancement in Jc is produced at moderate fluence levels (≈ 2-7 x 10^{10} cm^{-2}). Some datasets show even a six-fold increase. This enhancement can be attributed to the formation of efficient flux-pinning centres such as point defects or tiny defect clusters. It strengthens vortex immobilization. However, the trend is not strictly monotonic. At higher fluences (≥ 6 x 10^{10} cm^{-2}), the J_c gain becomes less consistent. And in some cases the J_c ratio decreases toward unity or below, suggesting the onset of defect saturation or partial damage to current-carrying pathways. This drop is consistent with the onset of excessive lattice disorder, partial amorphization or reduced intergranular connectivity, all of which can counteract the benefits of controlled defect introduction. At very low fluence ($< 1 \times 10^{10}$ cm^{-2}) , slight Jc improvement is present consistent with insufficient defect density for effective pinning Thus the graph highlights that Jc enhancement is not linearly dependent on fluence but there exists a key principle that Jc enhancement is maximized only within an optimal fluence window, beyond which damage becomes detrimental [28].

Changes in Bi-2212/Bi-2223 phase fractions across processing steps

Ion irradiation is not a stand-alone solution for achieving single-phase Bi‑2223 films. It lacks the chemical influence necessary to stabilize the high -Tc phase. It cannot convert mixed-phase films into single-phase material by itself without concurrent chemical doping and thermal annealing. Ion irradiation is most useful when it is part of a larger step-by-step processing strategy. It works best when combined with chemical doping (which helps stabilize the Bi- 2223 phase), careful film-growth methods (which set the initial structure), and post annealing (which repairs damage and activates helpful changes). Irradiation acts as a fine tuning tool that can adjust nanoscale defects and modify the local structure in ways that doping or deposition cannot. Additionally, if irradiation parameters such as ion species, fluence, and energy are not carefully optimized, the process can result in undesirable lattice damage, reduced crystallinity, and degradation of the superconducting transition temperature. Moreover, there is a need for specialized equipment such as particle accelerators and ion beam sources in addition to the technically complex process control. These factors make it difficult to use it widely in routine or large scale fabrication.

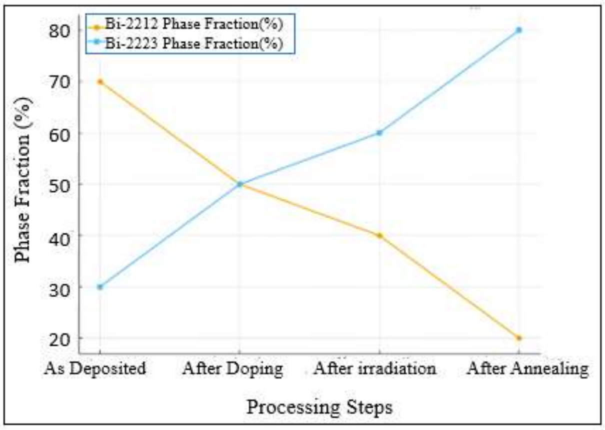

Fig 4: Phase- fraction evolution graph

The graph (Fig 4) illustrates how sequential hybrid processing steps, starting from as deposited, and progressing through doping, ion irradiation and post-irradiation annealing, affect the relative phase fractions of Bi-2212 and Bi-2223 in BSCCO thin films. As can be seen from the graph, Bi-2212 accounts for around 70% of the film in its as deposited state, while Bi-2223 makes up just about 30%. Chemical doping, particularly Pb substitution, leads to rise in Bi-2223 percentage to around 50%, indicating that doping stabilizes high T_c phase nucleation. Subsequent ion irradiation generates defect structures and boosts oxygen mobility, suppressing Bi-2212 and promoting Bi-2223 development to ~60%. The greatest substantial improvement occurs after annealing, when thermal activation restores irradiation- induced disorder and enables recrystallization and diffusion processes. At this stage Bi-2223 accounts for around 80% of the phase composition, while Bi-2212 contributes less than 20%. This trend show how combining chemical doping, irradiation and annealing can have a synergistic impact [29]. Irradiation produces defect assisted diffusion channels, chemical doping offers a favourable thermodynamic baseline and annealing promotes phase transition and recrystallization. These actions, taken together, gradually improves the superconducting performance and phase purity of the system.

Synergistic Strategies, Challenges and Future Directions

The most efficient approach to stabilize the Bi-2223 phase is to blend ion irradiation with other supportive techniques is. For instance, to grow the film in a more orderly manner and to reduce weak-link behaviour [30], Ion Beam Assisted Sputtering / Ion-Assisted Deposition (IAD) and use of textured substrates can be combined together. The chemical co-doping of Sb or rare earth ion (Yb, Er) in addition to Pb helps in stabilizing the Bi-2223 phase by modifying the charge balance. When these doped films are irradiated, the ion-induced defects and the dopants work together to enhance the phase purity and also increases the flux pinning. Other hybrid methods such as oxygen implantation followed by careful annealing can control oxygen levels precisely in the film. This is significant since phase stability and T_c are both strongly affected by oxygen content. However, more systematic research is still required to fully validate and optimize these combined approaches.

There are yet no reliable reports of complete single phase Bi-2223 films made only using irradiation-based techniques, despite significant advancements. The majority of studies highlight improvements in flux-pinning, defect control or partial suppression of Bi-2212. This suggests that while oxygen redistribution and defect engineering can benefit from irradiation, but obtaining pure Bi-2223 necessitates additional structural and chemical support.

More methodical research is necessary to advance this field by elucidating the ways in which particular ion factors like species, fluence and energy influence the phase formation. It is challenging to compare the outcomes or translate findings into scalable procedures due to the absence of standardized methodologies. Predictive control of phase behaviour is made impossible by the poor understanding of how irradiation induced defects evolve during post-annealing.

Implementation of advanced real-time measurement tools is required for future research work as traditional methods like XRD, TEM SQUID etc show how films appear only after the processing is complete. While, In-situ techniques like synchrotron grazing-incidence XRD, time resolved TEM during irradiation or real time, Raman spectroscopy can reveal what is happening during irradiation and annealing. These devices can monitor the movement of oxygen, defect migration and phase change in real time. With such information, researchers would be able to directly observe breakdown of Bi-2212 regions and beginning of formation of Bi-2223 under the ion beam. With this information processing conditions might be fine-tuned more precisely and consistently.

How defects and lattice strain that are created during ion irradiation contribute to formation of Bi-2223 phase is a major issue that needs more investigation. While too many defects can harm the lattice and destroy superconductivity, too few defects have no effect. To identify defect types, concentration and strain conditions that allow formation of Bi-2223 without harming material, future research should make use of focused experiments and computational techniques like DFT and molecular dynamics simulations.

In summary, the lack of reproducible single-phase Bi-2223 films shows how difficult it is to depend on ion irradiation alone. Irradiation works best when it is combined with other methods. Making this approach successful will require real-time monitoring, a better understanding of how defects help atoms move, and careful testing of irradiation settings. By combining ion beam research with advanced measurement tools and computer modelling, future studies may finally develop controlled ways to stabilize the Bi-2223 phase and possibly apply the same ideas to other complex oxide superconductors.

III. Conclusion

To conclude making thin films that contain only high Tc Bi-2223 phase is still very challenging. Normal methods help improve the amount of Bi-2223, but cannot fully remove the lower Tc Bi-2223 phase. Ion irradiation offers new ways to film’s structure, move oxygen and support the growth of Bi-2223. However, irradiation by itself has not yet produced fully single phase Bi-2223. The hybrid approaches help suppress Bi-2212, improve grain alignment and enhance superconducting performance. For real progress, future work must identify the best irradiation condition, study how defects help Bi-2223 grow and use real time tools to watch phase changes as they happen. If these challenges are addressed, ion irradiation could become a reliable part of producing high Tc Bi-2223 films for advanced superconducting technologies.

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