العدد 2 21A تموز 2026
المجلة العراقية للبحوث الانسانية والاجتماعية والعلمية
NO.21A2 Jul.2026 Iraqi Journal of Humanitarian, Social and Scientific Research
Print ISSN 2710-0952 Electronic ISSN2790-1254

تطويع خصائص التوصيل الفائق لمركب $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$: مراجعة نقدية لأليات التشويب بالرصاص ومنهجيات التخليق الكيميائي

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الملخص

يُعدّ طور Bi-2223 من نظام Bi-Sr-Ca-Cu-O (BSCCO) ذا أهمية بالغة نظرًا لارتفاع درجة حرارة التحول الحرجة (Tc~110 K) لاستخدامه في التطبيقات الصناعية عالية الطاقة. إلا أن تصنيع طور Bi-2223 النقيّ يُعدّ صعبًا بسبب المنافسة الشديدة مع طور Bi-2212 وضيق نطاق استقراره الديناميكي الحراري. في هذه الورقة، نستعرض بشكل شامل منهجيات التصنيع والدور الرئيسي لاستبدال الرصاص (Pb) في تثبيت بنية Bi-2223. ندرس تأثير Pb^{+2} على آلية الطور السائل العابر الذي يُحسّن حركية التفاعل عن طريق تثبيط التعديلات البنيوية. كما نُقدّم دراسة مقارنة بين تفاعل الحالة الصلبة (SSR) والطرق الكيميائية الرطبة (طريقة سول-جل، الترسيب المشترك)، وتأثيرها على مورفولوجيا الحبيبات وسلوك "الرابطة الضعيفة". كما تشير الدراسة إلى أن تحسين تركيز الرصاص (x ~ 0.3-0.4) ذو أهمية قصوى للحصول على أعلى كثافة تيار حرج (J_c) وتحسين البنية البلورية. وأخيرًا، تُناقش التوجهات المستقبلية للهندسة النانوية والتصنيع المستدام للموصلات الفائقة BSCCO، وذلك لتقديم خارطة طريق لأجيال مستقبلية من تكنولوجيا الموصلات الفائقة.

الكلمات المفتاحية: Bi-2223؛ تطعيم الرصاص، الموصلية الفائقة، طريقة سول-جل، استقرار الطور، كثافة التيار الحرج.

Tailoring the Superconducting Properties of $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$: A Critical Review of Lead-Doping Mechanisms and Synthesis Methodologies

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Abstract

Bi-2223 phase of the Bi-Sr-Ca-Cu-O (BSCCO) system is of high importance because of its high value of critical transition temperature (T_c~110 K) for use in high power industrial applications. The synthesis of phase-pure Bi-2223 is, however, difficult because of the close competition of the Bi-2212 phase and the narrow thermodynamic stability. In this paper, the synthesis methodologies and major role of Lead (Pb) substitution to stabilize Bi-2223 structure are comprehensively reviewed. We study the effect of Pb^{+2} on the mechanism of a transient liquid phase that enhances reaction kinetics by inhibiting structural modulations. A comparative study between Solid-State Reaction (SSR) and wet-chemical routes (Sol-Gel, Co-precipitation) is presented and its effect on the grain morphology and “weak-link” behavior. It also points out that the optimization of the concentration (x~ 0.3-0.4) of Pb is of paramount importance for obtaining maximum critical current density (J_c) and better texturing. Finally, the future directions of nano-engineering, and sustainable synthesis of

BSCCO superconductors are discussed to present a road-map for future generations of superconducting technology.

Keywords: **Bi-2223; Pb-doping, Superconductivity, Sol-Gel, Phase Stability, Critical Current Density.**

1. Introduction

One of the most daunting problems and greatest opportunities in the field of contemporary materials science is that of High-Temperature Superconductivity (HTS). Since the discovery of the Bismuth-based (Bi-Sr-Ca-Cu-O) system in 1988, the Bi-2223 phase, with the chemical formula $Bi_2Sr_2Ca_2Cu_3O_{10}$ has emerged as a cornerstone for clean energy technologies and large-scale industrial applications. This is mostly because of its high critical transition temperature (T_c) more than (110 K) that enables relatively inexpensive operation while providing liquid nitrogen as a coolant instead of the very expensive liquid helium [1, 2]. The structural complexity of the BSCCO family comes from the close thermodynamic proximity of three phases – Bi-2201 ($T_c \approx$ 20K), Bi-2212 ($T_c \approx$ 85K) and the technologically important but synthetically less desirable Bi-2223 ($T_c \approx$ 110K). The crystallography unit cell of the BSCCO crystalline structure is shown in figure 1 following [3].

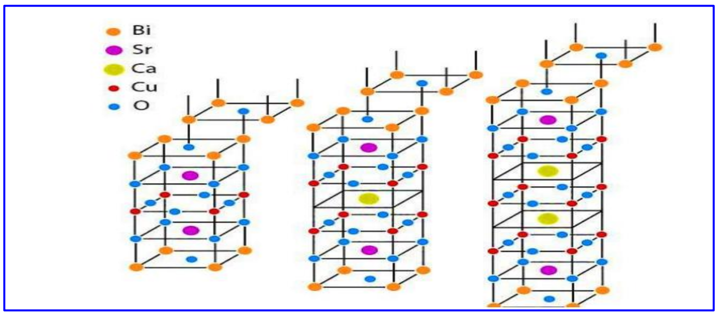

Figure1. The crystal unit cells of BSCCO family in different phases[3].

One of the problems in the fabrication process is the meta-stable nature of the 2223 phase it forms in a very small temperature window around the partial melting point and it is frequently fabricated as a secondary phase enclosed in the more stable 2212 matrix [4].

The use of Lead (Pb^{+2}) has changed the way these materials are processed. The lead is acting as structural stabilizer, substituting Bismuth (Bi^{+3}) sites in the insulating layers, extends the stability range of the 2223 phase and catalyzing its formation is facilitated by a lead-rich liquid phase sintering mechanism. Moreover, the Pb-doping is a key factor in improving the inter granular connectivity which is very important in overcoming the "weak-link" behavior that contributes to limit the high critical current densities (J_c) in poly-crystalline ceramic superconductors [56,]. The traditional Solid-State Reaction (SSR) technique has been the most commonly used one for bulk production, but the need for control of grain morphology and the chemical homogeneity has stimulated work on new "wet chemistry" methods.

Sol-Gel synthesis, Co-precipitation and Auto-combustion techniques are included. There are some pros for each method in terms of reaction kinetics, energy efficiency and the resulting superconducting properties [7]. In this review, the recent developments on the synthesis strategies of $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ are critically and comprehensively analyzed. The effects of processing parameters, including sintering temperature, annealing time and oxygen partial pressure on the basic superconducting properties are discussed. This paper compares conventional and non-conventional fabrication methods and thus determines the optimum route to high performance superconductors to satisfy the rigorous requirements of magnetic and electrical power applications of the next generation [8].

2. Mechanism of Lead (Pb) Substitution in Bi-2223 Phase

The most important factor for stabilizing high-Tc phase is the substitution of Bi^{+3} by Pb^{+2}. The following physical and chemical viewpoints can be used to explain the underlying mechanisms:

2.1. Liquid Phase Facilitation and Kinetics

The formation regarding Bi-2223 phase from Bi-2212 is diffusion controlled that is naturally slow. It has been found that Pb substitution causes a decrease in the solidus temperature of the system, thus favoring the formation of a transient liquid phase [9]. This liquid phase is often attributed to the melting of Ca_2PbO_4 and serves as a flux that promotes diffusion of Ca^{+2} and Cu^{+2} ions that are necessary to convert the double CuO_2 layer (2212) to the triple CuO_2 layer (2223) [10].

2.2. Structural Stabilization and Modulation Suppression

The incommensurate structural modulation in the Bi-O layers is one of the features of Bi-based superconductors. Ahadov, A. et al. have shown that these modulations can be suppressed by Pb ions, which can relax the internal lattice strain of the mismatch between the Bi-O and Cu-O_2 layers [11]. The stabilization is important as it results in a more uniform crystal structure and eliminates the stacking faults which are often observed in c-axis growth [12].

2.3. Optimization of Carrier Concentration (Hole Doping)

The hole concentration in the Cu-O_2 planes is crucial for the superconducting properties in cuprates. Replacing Pb^{+2} for Bi^{+3} causes a change in the oxygen content δ, thus tuning the charge carrier density [13]. Optimum Pb content (usually $x \approx 0.3$-0.4) place the system in the optimally doped region, where the critical temperature (T_c) is maximum, around 110 K, as demonstrated by the studies of Gayathri, V. et al. [14].

2.4. Enhancement of Grain Connectivity and J_c

The role of Pb from a micro-structural point of view is to promote the development of large plate-like grains and to enhance their orientation (texturing). This is essential to reduce the effect of "Weak-link" at the grain boundaries [15]. The coupling between superconducting grains is improved by Pb substitution, and results in a greatly increased

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transport critical current density (J_c), which is essential for industrial wire and tape applications [16].

3.Advanced Synthesis Methodologies of $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$

Bi-2223 is very sensitive to thermal history and chemical precursor homogeneity in the synthesis process. The major fabrication routes will be discussed below.

3.1. Conventional Solid-State Reaction (SSR)

The SSR method is the most technically simple method to be used as the benchmark for bulk production although it has the problem of phase purity.

- Precursor Processing: Stoichiometric amounts of high-purity (≥ 99.9%) Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, and CuO are pulverized. The use of carbonates requires a multi-stage calcination process (typically at 800-820 °C) to ensure complete CO_2 evolution, which is critical to avoid porosity [17].
- Thermal Cycles: The formation of the 2223 phase occurs via a long-term sintering process (100-250 h) at temperatures narrowly bracketed between 845 °C and 860 °C as shown in figure 2

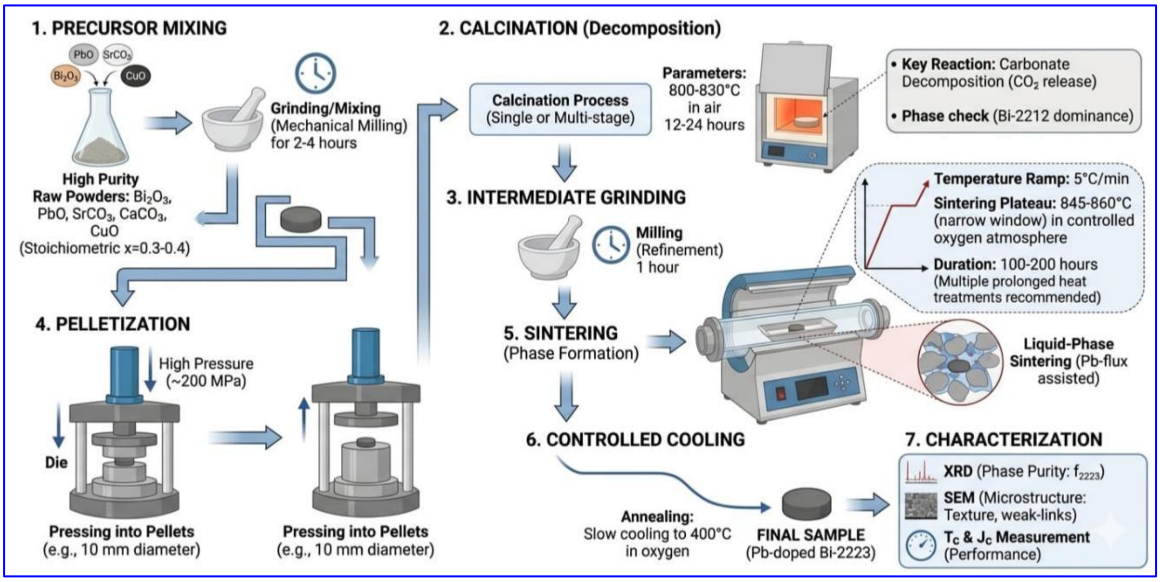

Figure 2. show flow chart of solid state reaction (SSR) synthesis for Pb- doped Bi-2223 ($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$)superconductor.

- Critical Drawback: SSR often results in chemically in homogeneous samples with residual Bi-2212 and Ca_2PbO_4 phases at grain boundaries, which act as scattering centers for Cooper pairs [18].

3.2. Sol-Gel Chemical Route (Polymeric Precursor Method)

To overcome the limitations of SSR, the Sol-Gel technique is employed to achieve atomic-level mixing.

- Mechanism: Metal nitrates are typically dissolved in a chelating agent such as Citric Acid or EDTA. The formation of a stable metal-chelate complex prevents the selective precipitation of individual components as shown in figure 3.

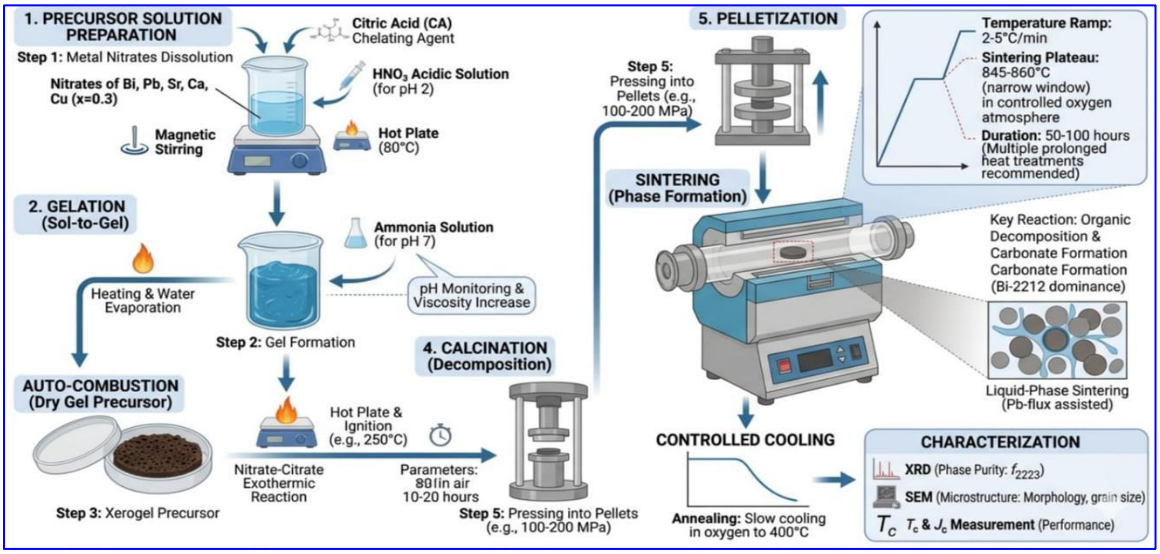

Figure 3. show flow chart of sol- gel (citrate - nitrate) synthesis for Pb- doped Bi-2223 ($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$)superconductor.

- Advantages:

1. Lower Sintering Temperature: The high surface area of the resulting nano-crystalline powder reduces the activation energy for Bi-2223 formation [19].

2. Enhanced Connectivity:* Sol-gel derived samples exhibit superior grain alignment and a significant reduction in the "Weak-link" effect compared to SSR.

- Quantitative Metric: Studies indicate that Sol-Gel samples can reach 95% phase purity in less than 50 hours of sintering, a 75% reduction in processing time compared to SSR [20].

3.3. Co-precipitation and Self-Combustion Methods

These methods are based on fast kinetics and very small particle size.

- Co-precipitation: Using oxalates or carbonates to precipitate all cations in simultaneous way. This ensures a highly reactive precursor but requires stringent pH control to maintain stoichiometry.

- Solution Combustion: This involves an exothermic reaction between an oxidizer (nitrates) and a fuel (glycine or urea). The sudden release of heat and gases produces highly porous, friable "fluffs" with a high density of flux-pinning centers, which is vital for high-field applications [21] as shown in figure 4.

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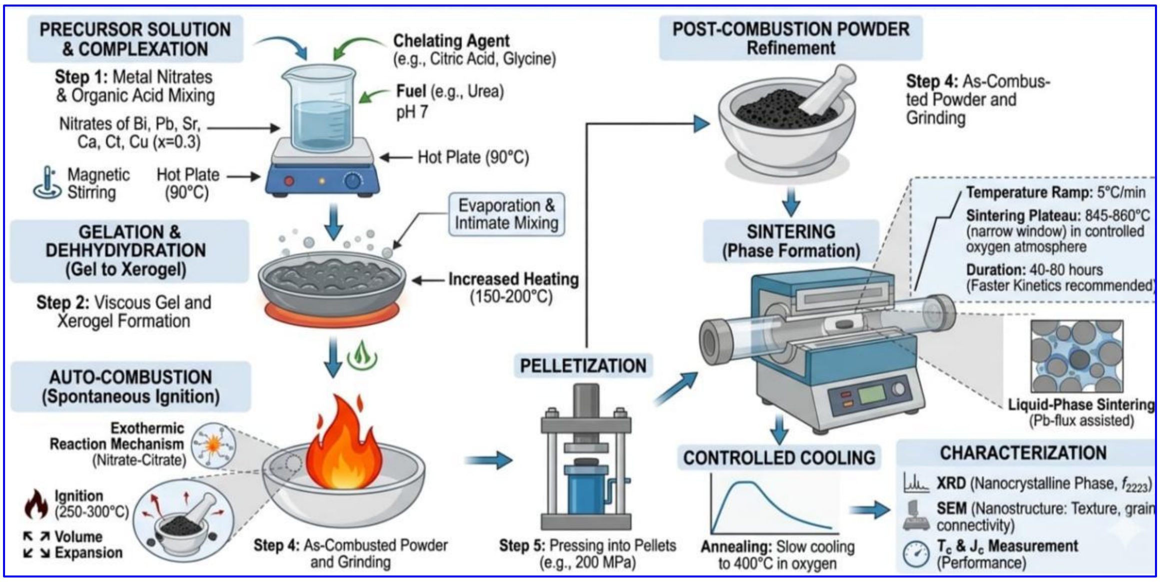

Figure 4. show flow chart of organic- acid facilitated auto - combustion synthesis for Pb- doped Bi-2223 ($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$)superconductor.

It is necessary to have a comparative analysis of the fabrication techniques in order to have a complete picture of the methods discussed and to make a comparison of their respective efficiencies. The main difference between the conventional Solid-State Reaction (SSR) and the different chemical routs (Sol-Gel and Co-precipitation) is summarized in Table 1. A comparison with critical parameters such as the homogeneity of the precursor, sintering duration and the purity of the resultant phase is made, and a clear road-map for choosing the optimum synthesis method for the desired superconducting performance is provided.

Table 1: Summary of synthesis parameters and performance metrics for various fabrication methods of Bi-2223.

Parameter	Solid-State (SSR)	Sol-Gel	Co-precipitation
Precursor Homogeneity	Low (Micron scale)	Excellent (Atomic scale)	High
Sintering Time	100-200 hours	24-60 hours	40-80 hours
Grain Size	Large (5-20 µm)	Sub-micron(200-800 nm)	Fine (1-3 µm)
Phase Purity	Moderate (70-85%)	Very High (>95%)	High (>90%)
Jc Potential	Low to Moderate	High	Moderate to High

4. Physical Characterization and Performance Metrics

A multi-faceted analytical approach is needed to correlate the micro-structural features with the macroscopic superconducting properties to validate a successful $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ synthesis.

4.1. Phase Identification via X-Ray Diffraction (XRD)

XRD is most commonly used for quantification of 2223/2212 phase ratio.

- Peak Analysis: The presence of Bi-2223 phase is confirmed by the sharp (00<u>10</u>) and (00<u>12</u>) reflections at $2\theta \approx 24°$ and 29°, respectively.
- Lattice Parameters: Usually, substitution of the Pb causes a slight increase in a and b axes and contraction in c axis, which is the structural change that allows higher Tc stability [22].

4.2. Morphological and Microstructural Analysis (SEM/EDX)

The grain growth dynamics can be understood by Scanning Electron Microscopy (SEM).

- Grain Morphology: The high quality Pb doped samples should have the plate-like or mica-like morphology. These plates (texturing) are aligned to give a direct indication of the current-carrying capacity.
- EDX Mapping: Energy Dispersion X-ray (EDX) spectroscopy is used to confirm the stoichiometric distribution of Bi, Pb, Sr, Ca and Cu. Any segregation of Lead-rich secondary phases (such as Ca_2PbO_4) at grain boundaries is presumed to be a precursor to the "weak-link" problem which is detrimental to the Jc [23].

4.3. Magnetic and Electrical Transport Measurements

The final evidence of superconductivity is the transition metrics:

- Temperature Dependence of Resistivity (ρ-T): Sharp single-step drop to zero to zero resistivity at the Tc~ 105-110 K which is indicative of high purity 2223 phase. The transition is a “double-step” one, which means it is a mixed-phase system (2212 + 2223) [24].
- AC Susceptibility: This would allow one to separate out the intrinsic superconductivity (within grains) and the extrinsic coupling (between the grains). Important parameter to probe energy dissipation and strength of flux pinning is the imaginary part (χ'') of susceptibility.
- Critical Current Density (Jc): The data of Jc at 77K in different magnetic field are essential to assess the material's use for industrial power applications [25].

5. Discussion: The Correlation between Processing and Performance

The data of this section suggests that there is an important synthesis of data. It should be observed that although doping with Pb is essential to obtain the desired liquid phase for 2223 growth, the cooling rate after sintering is also very important. Fast cooling will enable the preservation of the meta stable higher temperature phases while slow cooling in an oxygen-rich environment will enable better optimization of the oxygen non-stoichiometry δ and the hole doping state of the CuO_2 planes [26].

6. Conclusion and Future Outlook

The synthesis as well as characterization regarding $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ system is a very important interface between fundamental solid state chemistry and advanced cryogenic engineering. The multi-dimensional effect of Lead (Pb) substitution on phase stability and electromagnetic performance of the BSCCO system has been systemically analyzed in this review.

6.1. Key Findings

- Phase Stability: It is concluded that the substitution of Pb is also needed for the practical realization of Bi-2223 phase. The introduction of transient liquid phase, however, Lead overcomes the thermodynamic "bottlenecks" that typically favor the lower-Tc Bi-2212 phase.
- Morphological Synergy: The shift in the traditional Solid-State Reaction (SSR) to chemical routes such as Sol-Gel and Co-precipitation has proved to be a time saving measure and has also shown considerable improvement in the grain texturing. These methods enable the production of dense micro-structures with plate-like configuration, which is crucial to reduce the "weak-link" effect.
- Electronic Optimization: The substitution of Pb^{+2} at Bi^{+3} sites can be used as a fine tuning mechanism to control the hole doping level to achieve the optimum hole doping level of $Cu\text{-}O_2$ planes for a Tc of 110 K.

6.2. Future Directions

Despite the maturity of BSCCO research, several frontiers remain open for high-impact investigation:

1. Nona-Engineering: Incorporating artificial pinning centers (such as carbon nanotube or oxide nana-particles) into the Pb-doped matrix to enhance Jc under high external magnetic fields.

2. Sustainable Synthesis: Developing Green Chemistry protocols to reduce the environmental footprint and energy consumption of the long-term sintering processes.

3. Hybrid Composites: Exploring the interface between Bi-2223 and 2D materials (like Graphene or MoS_2) to improve the mechanical flexibility of superconducting tapes without compromising their electrical integrity.

In summary, while the Bi-2223 phase has reached a level of industrial readiness, the optimization of its synthesis via Pb-doping continues to provide a fertile ground for achieving the theoretical limits of high-temperature superconductivity.

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