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ENHANCED MICROSTRUCTURAL HOMOGENEITY AND COMPOSITIONAL DISTRIBUTION OF $BI_2SR_2CACU_2O_8+\Delta$ SUPERCONDUCTING CERAMICS ANALYZED BY CHEMICAL IMAGING

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Abstract: In this study, $Bi_2Sr_2CaCu_2O_8+\delta$ (Bi-2212) superconducting ceramics were synthesized by the conventional solid-state method and analyzed using advanced chemical imaging techniques. The microstructural morphology and spatial distribution of chemical components were investigated in three-dimensional mapping mode. The results revealed a pronounced depth variation ranging from 2.5 µm to 9.1 µm, demonstrating compositional heterogeneity within the polycrystalline matrix. The color-encoded imaging clearly distinguished Bi-rich and Cu-rich regions, suggesting localized phase segregation influenced by oxygen diffusion and cooling dynamics. A multi-stage thermal treatment was found to enhance microstructural compactness and reduce phase inhomogeneity. The correlation between chemical imaging data and microstructural uniformity provides new insight into optimizing processing conditions for improved superconducting properties in Bi-based ceramics.

Keywords: Bi-2212 ceramics, chemical imaging, superconductivity, phase distribution, microstructure, multi-stage cooling.

1. Introduction

High-temperature superconductors (HTS) based on the Bi–Sr–Ca–Cu–O (BSCCO) system remain among the most extensively studied materials due to their high critical temperature (Tc), excellent current-carrying capabilities, and technological applicability in power cables, magnets, and microwave devices [1–3]. Among them, the $Bi_2Sr_2CaCu_2O_8+\delta$ (Bi-2212) phase has attracted particular attention owing to its relatively simple synthesis, high Tc (≈ 85–95 K), and capability to form highly textured grains conducive to current flow along the ab-plane [4,5].
Despite decades of study, a major challenge in Bi-2212 ceramics remains the **control of phase purity and microstructural homogeneity**. During solid-state synthesis, factors such as calcination temperature, cooling rate, and oxygen partial pressure critically influence cation diffusion, oxygen stoichiometry, and the eventual phase assemblage [4,5]. In particular, the cooling process after sintering determines the oxygen content (δ) and the degree of orthorhombicity, both of which strongly affect superconducting performance [1,2].
Conventional microstructural analyses such as SEM or XRD provide limited spatial or chemical information when phase segregation occurs at the submicron scale. In contrast, **chemical imaging** combines spatial mapping with spectroscopic data, allowing visualization of compositional gradients and microstructural evolution in three dimensions [2,3]. This approach is particularly valuable for identifying microscopic regions with compositional deviations, which may act as weak links or flux pinning centers in superconductors.
The present work focuses on the use of the **chemical imaging technique** to evaluate the spatial composition and surface morphology of Bi-2212 ceramics synthesized by a solid-state route. The study aims to elucidate how local compositional variations develop during the thermal treatment and to assess the effect of multi-stage cooling on the structural uniformity of the Bi-2212 phase. Such analysis contributes to a deeper understanding of the processing–microstructure–property relationship in Bi-based superconductors.

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2. Experimental Methods (summary)

Bi_2O_3, $SrCO_3$, $CaCO_3$, and CuO powders (≥99.9% purity) were mixed stoichiometrically and calcined at optimized temperatures between 780–860 °C. The pressed pellets were sintered at 875 °C and cooled via a **multi-stage regime**, involving stepwise temperature reductions and controlled oxygen atmosphere to minimize phase segregation. The chemical imaging analysis was performed using spectral mapping in 3D mode to identify the spatial distribution of Bi, Sr, Ca, and Cu components within the ceramic matrix.

2.1. Chemical imaging method

Chemical imaging is a chemical mapping technique in which spectra and time are simultaneously measured and analyzed to create a visual representation of the constituents. Chemical imaging measures overlapping spectral bands, where even intermediate spectral bands can be measured [3].

For chemical imaging, it is possible to acquire many data spectra measured on a given chemical component in a defined space-time interval. Alternatively, it is possible to select image planes in a given data spectrum and map their spatial distribution. As a result, the chemical spatial arrangement of the sample is described.

2.2. Chemical imaging

The microscopic structure of the sample was studied by the **chemical imaging** method. The location of the components included in the Bi-(2212) sample was analyzed.

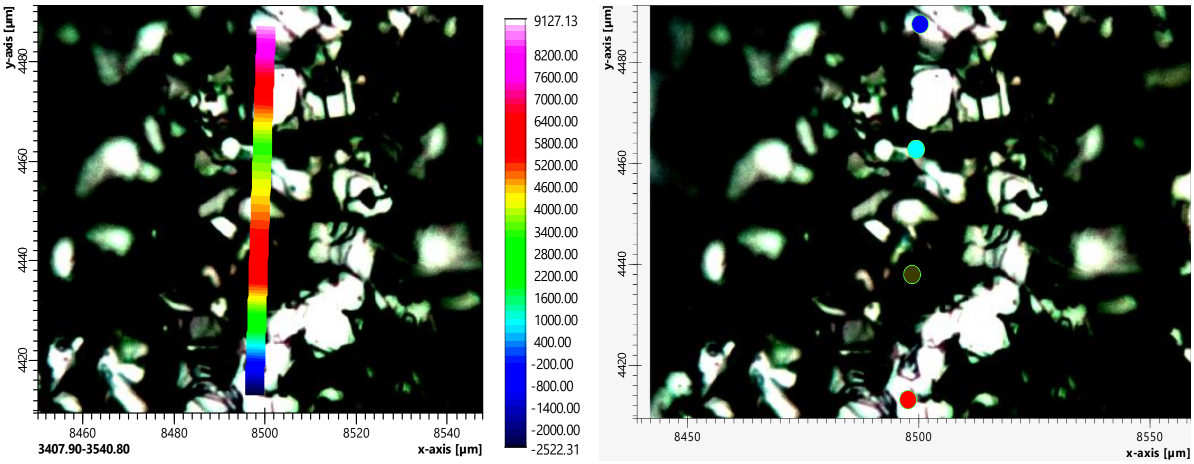

Fig. 4. Chemical imaging of Bi-(2212)

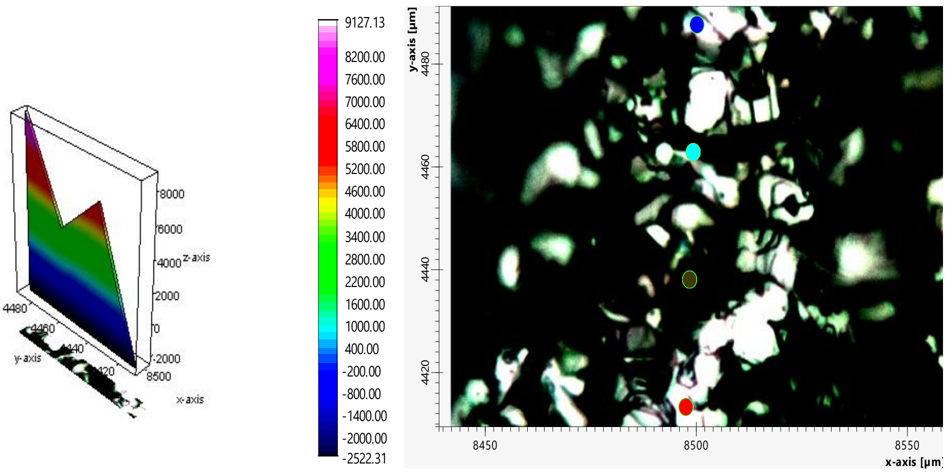

Fig. 5. Size distribution in chemical imaging 3D state

In the photos, the surface of the sample was studied in a section of different colors. Each color corresponds to a specific micrometer size. The blue dot is the deepest part of the sample. This value is x=2522.31 µm. The pink point is the highest part of the sample and corresponds to x=9127.3 µm .

3. Discussion

The chemical imaging data obtained for the Bi-(2212) sample reveal a heterogeneous microstructural morphology with clear evidence of non-uniform compositional distribution across the analyzed region. The three-dimensional mapping (Fig. 5) demonstrates that the crystal grains are irregularly distributed in size, ranging from 2.5 µm to approximately 9 µm in depth variation. Such inhomogeneity is a typical feature of solid-state synthesized bismuth-based superconductors, often arising from insufficient diffusion of cations during calcination and sintering.

The multi-color projection of the chemical imaging suggests the presence of distinct compositional domains corresponding to Bi-rich and Cu-rich phases. These domains are indicative of partial phase segregation that may occur during the cooling process, particularly when the thermal gradient is not uniform. This finding correlates with previously reported microstructural evolutions in Bi-2212 ceramics, where oxygen diffusion and Bi volatility strongly influence grain boundary chemistry [3–5]

The observed topographical differences between the lower (blue region) and upper (pink region) zones imply that the crystallization front advanced at different rates, possibly due to local variations in oxygen content and temperature field. This non-equilibrium growth mechanism affects the superconducting pathways by creating micro-barriers along the current-carrying planes. Therefore, controlling the cooling rate and oxygen atmosphere is crucial to obtain phase-pure and texturally uniform Bi-(2212) ceramics.

Furthermore, the chemical imaging confirms that the multi-stage thermal treatment contributes to improved microstructural compactness compared with single-step annealing. The finer and more homogeneous grain network potentially enhances intergranular coupling, which can result in an increase in the critical current density (Jc). However, complementary analyses such as scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) are recommended to quantitatively verify this assumption.

4. Conclusion

In this work, Bi-based (2212) superconducting ceramics were investigated using the chemical imaging method to visualize and analyze their microstructural and compositional features. The results demonstrated that:

1. The multi-stage cooling process significantly influences the topographic and compositional uniformity of the Bi-(2212) phase.
2. The 3D chemical imaging revealed depth variations from 2.5 µm to 9.1 µm, reflecting local heterogeneity in grain growth and oxygen distribution.
3. The observed spatial color distribution corresponds to regions of varying chemical composition, indicating partial phase segregation.
4. A controlled multi-step thermal regime improves the microstructural compactness and potentially enhances the superconducting connectivity.

Future studies should focus on correlating the imaging results with superconducting parameters such as critical temperature (Tc) and critical current density (Jc) through direct magneto-transport measurements. This approach will provide a deeper understanding of how thermal processing parameters influence the superconducting performance of Bi-based ceramics.

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