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Article.)

Influence of Sintering Temperature on the Transport Properties of $\text{GdBa}_2\text{Cu}_3\text{O}_7$ Superconductor Prepared from Nano-Powders via Co-Precipitation Method

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Abstract

This study examines the influence of sintering temperature on the structural and transport properties of $\text{GdBa}_2\text{Cu}_3\text{O}_7$ (Gd123) superconductors prepared from nano-sized precursors via the co-precipitation method. The metal-oxalate precursor (average particle size <50 nm) was calcined at 900°C for 12 hours, then the prepared pellets were sintered in oxygen at 920 – 950°C for 15 hours. All samples showed metallic properties and a sharp superconducting transition. Critical temperatures $T_{\text{C}(R=0)}$ were 94 – 95 K, with higher sintering temperatures steadily boosting critical current density. X-ray diffraction confirmed orthorhombic Gd123 as the dominant phase, with its phase fraction increasing from 92% to 99.8% as the sintering temperature increased. SEM micrographs showed large, densely packed grains, with higher sintering temperatures promoting improved grain connectivity and reduced porosity. The sample sintered at 950°C exhibited the most favorable transport performance, attributed to enhanced intergranular coupling and the presence of nanoscale secondary phases acting as effective flux-pinning centers. Overall, these results demonstrate that careful control of sintering temperature can significantly optimize the microstructure and superconducting properties of Gd123 materials, supporting their advancement for practical electrical and magnetic applications.

Keywords: $\text{GdBa}_2\text{Cu}_3\text{O}_7$; superconducting ceramic; co-precipitation method; electrical resistance; critical current density; crystal structure

1. Introduction

The synthesis of $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE123) ceramic superconductors has attracted considerable interest due to their promising electrical and magnetic properties [1–8]. Various synthesis methods have been employed, including the conventional solid-state reaction (SSR) [4], pulsed laser deposition (PLD) [9], co-precipitation (COP) [10–15], and chemically modified photosensitive techniques [16–17]. The SSR method typically uses high-purity oxides and carbonates as precursors, involving multiple grinding steps and extended heat treatments to ensure complete reaction. However, this approach can introduce contamination during processing [18–20]. In contrast, the COP

method offers advantages by employing nanoscale precursors, reducing the need for prolonged sintering and grinding [21–24].

Among rare-earth RE123 compounds, gadolinium-based $\text{GdBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (Gd123) exhibits an orthorhombic crystal structure at low oxygen deficiency ($\delta < 0.6$) and a critical temperature (TC) exceeding 90 K [7]. The presence of secondary phases such as $\text{Gd}_2\text{BaCuO}_5$ (Gd211) has been reported to enhance transport properties by acting as flux pinning centers [8].

Previous studies have established that sintering time affects Gd123 superconducting performance, with an optimal duration of 15 hours at 920 °C identified [10–15]. Nonetheless, how changes in sintering temperature affect the superconducting and transport properties of Gd123 produced by COP has not been thoroughly investigated.

This study examines how sintering temperature affects phase formation, microstructure, and transport properties in Gd123 ceramics made by coprecipitation. Analyses included XRD, TGA, DC electrical resistance tests, and SEM.

2. Materials and Methods

2.1 Materials

Gadolinium(III) acetate tetrahydrate $\{\text{Gd}(\text{CH}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}\}$, barium acetate $\{\text{Ba}(\text{CH}_3\text{COO})_2\}$, and Copper(II) acetate monohydrate $\{\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}\}$, were purchased from Sigma Aldrich, supplied with a purity greater than 98%, and used as received. Oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$), isopropanol ($\text{C}_3\text{H}_8\text{O}$), and distilled water were used as required.

2.2. Superconductor Powder Synthesis

A clear aqueous solution of a mixture of 6.92 mmol $\text{Gd}(\text{CH}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$, 13.85 mmol $\text{Ba}(\text{CH}_3\text{COO})_2$, and 20.78 mmol $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ was prepared, and then 300 mL of 0.5 M oxalic acid and a 1.5 isopropanol: 1 water mixture were added for superconductor powder synthesis (Figure 1). The result was a stable blue suspension product that was separated and dried overnight, gaining a blue powder, as reported in the ref. [11]. The reaction equations are as follows:

2.3. Compaction and Sintering

The blue powder obtained was heated at 900 °C for 12 hours in a muffle furnace (Nabertherm, Germany), then cooled to room temperature at a rate of 2 °C per minute. After calcination, the powder was ground for 10 minutes using an agate mortar and pestle, then pressed into pellets about 1.25 cm in diameter with a Carver pressing machine (USA) at a compaction pressure of 300 MPa. These pellets were sintered at 920, 930, 940, and 950 °C under an oxygen flow of 5 mL/min for 15 hours in a tube furnace (Nabertherm, Germany), followed by slow cooling to room temperature at a rate of 1 °C per minute.

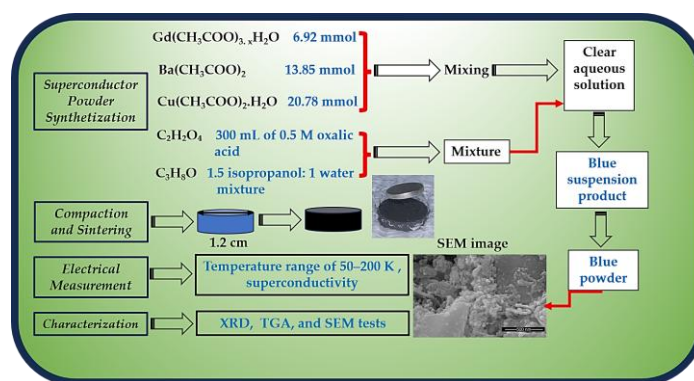


Figure 1. Schematic of research implementation.

2.4. Electrical Resistance Measurement

Electrical resistance measurements for the sintered pellets were conducted over a temperature range of 50–200 K using a standard four-probe method. A direct current (DC) of 30 mA was applied, and the experiments were carried out in a closed-cycle refrigerator system model ARS-EA202A (USA).

2.5. Characterization

Some of the sintered pellets were milled, and their powders were examined using X-ray powder diffraction (Malvern Panalytical, Aeris, monochromatic $\text{Cu K}\alpha_1$, 1.5406 Å, 0.02 step angle, with 2θ ranging from 4° to 60°, Almelo, The Netherlands) for phase and unit cell investigation. TGA analysis of the synthesized blue powder was performed on a Netzsch STA 409 PG/PC thermal analyzer (Selb, Bavaria, Germany) to determine weight loss as a function of temperature. SEM (FEI QUANTA 200, Netherlands) and (Hitachi S 3400 N, Japan) were employed for microstructural investigations of the produced blue superconductor powder and sintered pellets.

3. Results and Discussion

3.1. TGA Curve of The Produced Blue Nano-powder

The TGA curve showed five significant weight loss drops as a function of temperature, as illustrated in Figure 2. The first drop was the moisture loss from the sample, which ended at approximately 125 °C. The second drop, occurring at 125–226 °C, represented the water loss due to crystallization from the metal oxalate mixtures. The third drop in weight at 226–293 °C could be due to the decomposition of CuC_2O_4 , BaC_2O_4 , and $\text{Gd}_2(\text{C}_2\text{O}_4)_3 \cdot 6\text{H}_2\text{O}$ into CuO , BaCO_3 , and $\text{Gd}_2(\text{C}_2\text{O}_4)_3$. The fourth drop is associated with the decomposition of $\text{Gd}_2(\text{C}_2\text{O}_4)_3$ to Gd_2O_3 , and formation of BaCuO_2 and which agrees with previous reports [24, 25]. The final drop shows a complete decomposition and the formation of $\text{GdBa}_2\text{Cu}_3\text{O}_{7-x}$ that begins at 900 °C, as indicated in Table 1. Therefore, from the TGA thermogram, it can be concluded that the suitable calcination and sintering temperatures for Gd123 are in the range of 900–950 °C

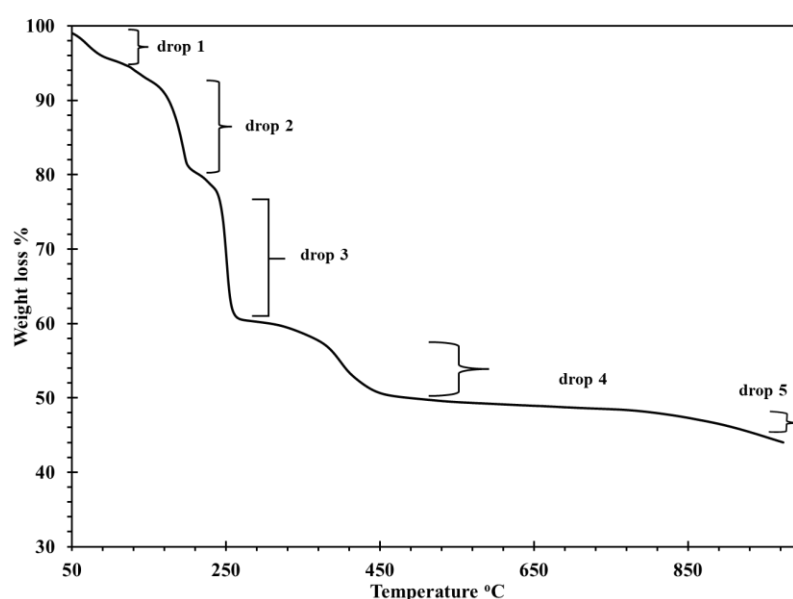


Figure 2. TGA curve of synthesized nanoscale powder.

Table. 1. Different stages of the reaction mechanism during the TGA test.

Weight losses (drops)	Reaction mechanism
Drop 1	Moisture loss from the surface of metal oxalate complex
Drop 2	$\text{BaC}_2\text{O}_4 \cdot 0.5\text{H}_2\text{O} \rightarrow \text{BaC}_2\text{O}_4 + 0.5\text{H}_2\text{O}$
	$\text{CuC}_2\text{O}_4 \cdot \text{H}_2\text{O} \rightarrow \text{CuC}_2\text{O}_4 + \text{H}_2\text{O}$
Drop 3	$\text{Gd}_2(\text{C}_2\text{O}_4)_3 \cdot 6\text{H}_2\text{O} \rightarrow \text{Gd}_2(\text{C}_2\text{O}_4)_3 + 6\text{H}_2\text{O}$
	$\text{BaC}_2\text{O}_4 \rightarrow \text{BaCO}_3 + \text{CO}$
	$\text{CuC}_2\text{O}_4 \rightarrow \text{CuO} + \text{CO}_2 + \text{CO}$
Drop 4	$\text{Gd}_2(\text{C}_2\text{O}_4)_3 \rightarrow \text{Gd}_2\text{O}_3 + 3\text{CO}_2 + 3\text{CO}$
	$\text{CuO} + \text{BaCO}_3 \rightarrow \text{BaCuO} + \text{CO}_2$
Drop 5	$0.5 \text{ Gd}_2\text{O}_3 + 2 \text{ BaCuO}_2 + \text{O}_2 \rightarrow \text{GdBa}_2\text{Cu}_3\text{O}_7 + 0.5 \text{ O}_2$

3.2. Microstructure of The Produced Powder and Sintered Pellets

The SEM image of the produced blue powder confirms the precipitation of nanoparticles, as shown in Figure 3. The image reveals the agglomeration state of the nanoscale particles, which is a probable result because of the mutual diffusion between particles during the precipitation process, in the absence of any capping substance. The particle size is within the range of 50 nm or less.

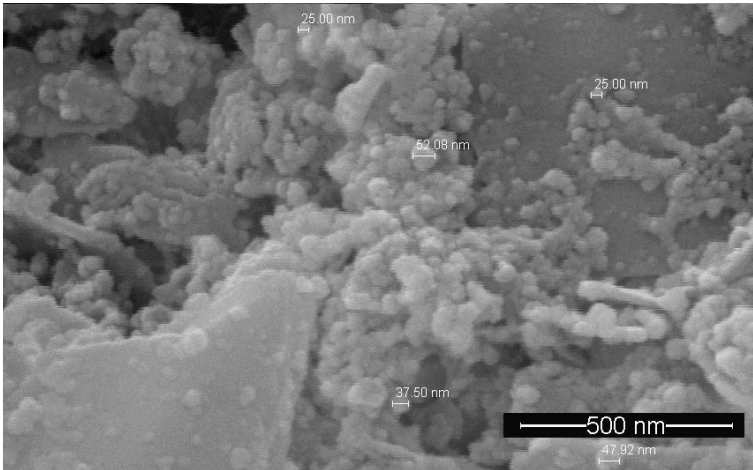


Figure 3. SEM image of the blue produced powder.

All sintered pellets exhibited large, highly compacted grains with melt-like structures, as demonstrated in Figure 4. In addition, the grains become closer and more coherent as the sintering temperature increases, thus improving the sintering density as a common result, which is in good agreement with the porosity and relative density measurements recorded in Table 3.

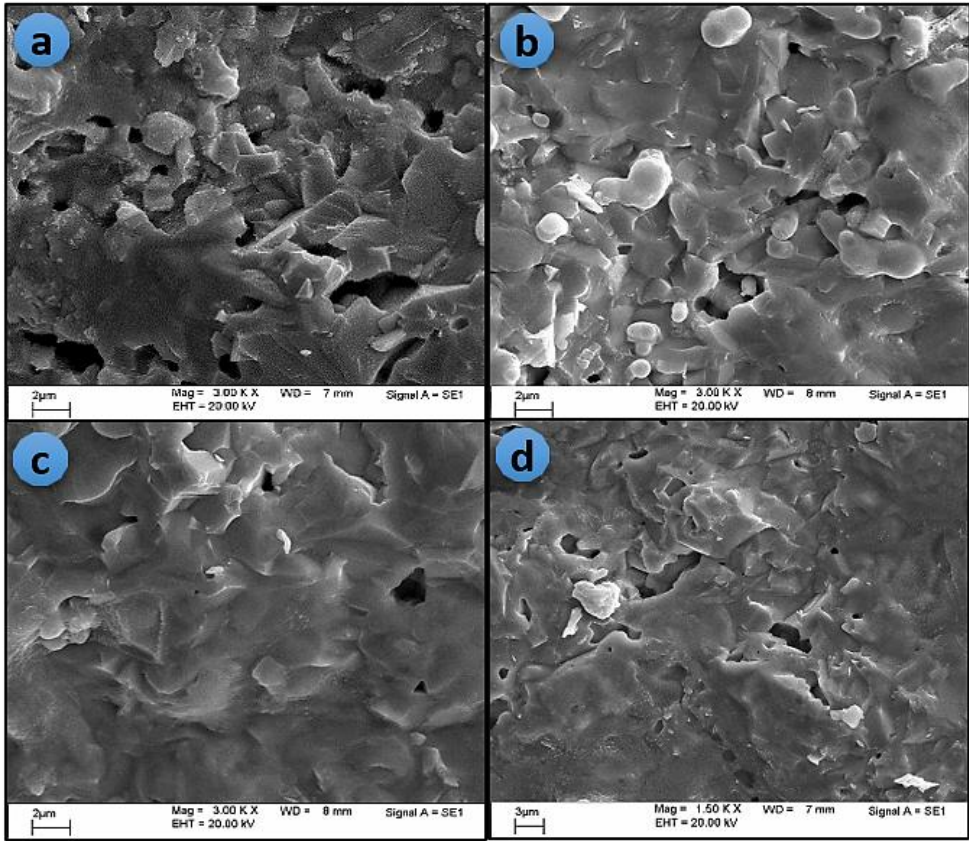


Figure 4. SEM images (SEI) of sintered pellets fractured surfaces, (a) the of sintered sample at 920 °C, (b) the image of sintered sample at 930 °C, (c) the image sintered sample at 940 °C, and (d) the image of sintered sample at 950 °C.

3.2. XRD Results

Figure 5 presents XRD patterns for sintered Gd123 at temperatures ranging from 920°C to 950°C. The dominant phase of the orthorhombic structure (Gd123) is presented for all samples. This phase was identified and labeled as (h k l) planes and the perovskite structure with the space group of Pmmm, No. 47, Z = 1, $\alpha = \beta = \gamma = 90^\circ$. The measured lattice parameters for all samples match the standard (JCPDS, # 01-089-5733) listed in Table 2. Few peaks belonging to impurities at 28.42°, 29.27°, 30.08°, 35.68°, 39.99°, 41.87°, 42.54°, and 49.27° were also detected and belonged to the non-superconducting phases, such as BaCuO₂ (Gd011) and Gd₂BaCuO₅ (Gd211).

GdBa₂Cu₃O_{7-δ} "Gd123%" phase contents for samples sintered at 920 °C, 930 °C, 940 °C, and 950 °C were 92.5%, 99.1%, 99.5%, and 99.8%, respectively. Impurities at 920 °C result from incomplete sintering, while 930–950 °C yields high Gd123 purity.

Table 2. Lattice parameters and volume for all sintered samples .

Sintering (°C)	a (Å)	b (Å)	c (Å)	Volume (Å)
920	3.849 ± 0.001	3.901 ± 0.001	11.718 ± 0.002	175.92± 0.02
930	3.846 ± 0.001	3.902 ± 0.001	11.716 ± 0.001	175.80± 0.03
940	3.842 ± 0.001	3.903 ± 0.001	11.722 ± 0.001	175.81± 0.02
950	3.843 ± 0.001	3.904 ± 0.001	11.723 ± 0.001	175.85± 0.02

The Gd123% was calculated using the following Equation (1) [11]:

$$\text{Gd 123 \%} = \frac{\Sigma \text{I}_{123}}{\Sigma \text{I}_{123} + \Sigma \text{I}_{\text{Impurities}}} \times 100\% \quad (1)$$

where I_{123} and $I_{\text{Impurities}}$ are the peak intensities of the observed phases for Gd 123 and impurities.

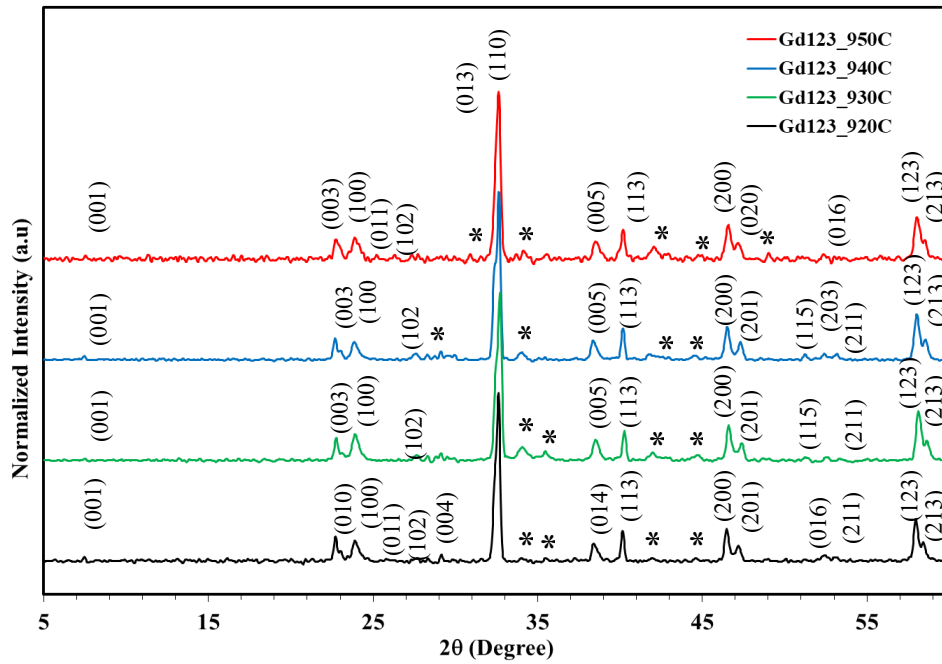


Figure 5. XRD patterns of Gd123 at different sintering temperatures. (h k l): Gd123, *: impurity phases.

3.4. Electrical Measurements

The normalized electrical resistance as a function of temperature, in the absence of a magnetic field, was measured for all sintered samples and showed normal metallic behavior with a single-drop transition feature, as shown in Figure 6. All sintered samples showed a zero-resistance temperature ($T_{C, R=0}$) of 94–95 K, with no notable changes as the sintering temperature increased.

The transport critical current density, J_c , was calculated using Eq. (2):[13]

$$J_c = \frac{I_c}{A} \quad (2)$$

where I_c represents the maximum current (A) through the sample's cross-sectional area (cm^2) before superconductivity is lost at 77 K and zero magnetic field. Figure 7 presents J_c values of 4.35 ± 0.11 , 6.35 ± 0.43 , 7.89 ± 0.71 , and 12.9 ± 0.89 A/ cm^2 for samples treated at 920, 930, 940, and 950 °C, respectively.

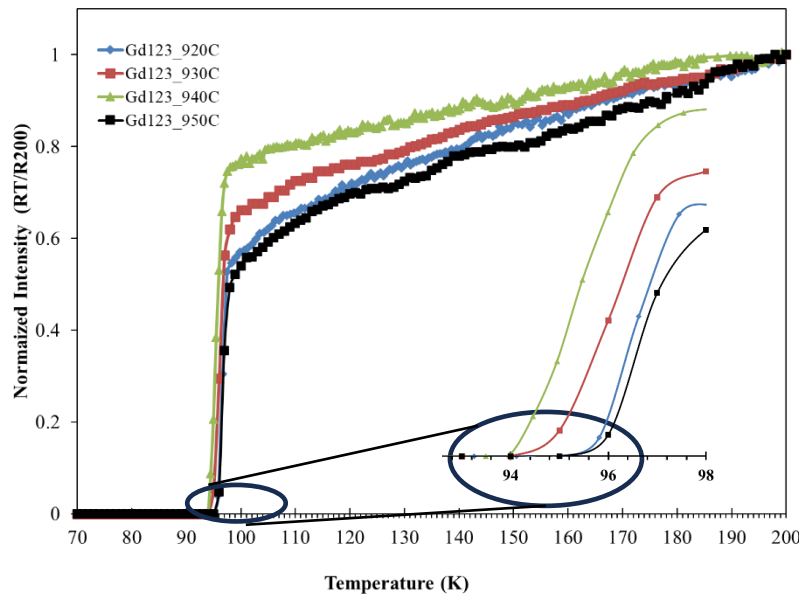


Figure 6. Normalized resistance of Gd123 as a function of temperature.

The J_c values presented in Table 3 (section 3.4) increased with higher sintering temperatures, attributed to a reduction in weak link formation at grain boundaries, thereby facilitating improved current flow. Additionally, the small quantity of 211 phases identified within the 123 superconducting system contributed positively to transport properties, as corroborated by XRD data (which indicates these phases are not superconductors). These 211 phases serve as flux pinning centers and may enhance current flow between grains, as discussed in references [15, 16, 25].

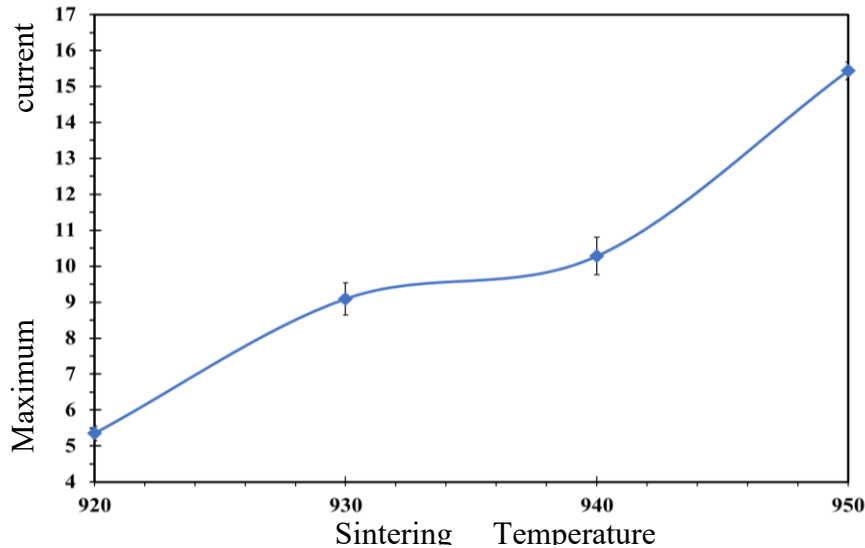


Figure 7. Maximum current density (J_c) versus sintering temperature.

3.5. Relative Density and Porosity

The relative density (D_{relative} %) and the porosity for all samples were calculated from the actual and theoretical densities using the following equations (3 & 4):

$$D_{\text{Relative}} \% = \frac{\rho_{\text{sample}}}{\rho_{\text{theoretical}}} \times 100\% \quad (3)$$

$$\text{Porosity \%} = \frac{\rho_{\text{theoretical}} - \rho_{\text{sample}}}{\rho_{\text{sample}}} \times 100\% \quad (4)$$

where the relative density of sample D_{Relative} is given by the percentage ratio of the density ρ_{sample} , obtained for the bulk samples, to theoretical density $\rho_{\text{theoretical}}$ obtained from the XRD data (Table 3). Increasing the relative density of ceramics plays a pivotal role in improving their critical current density. This enhancement is primarily attributed to the reduction of insulating cavities within the material, which facilitates better current flow between the grains.

Table 3. Summarized data of $T_{C(R=0)}$ (K), $T_{C\text{-onset}}$ (K), Gd123%, relative density, porosity and the maximum current density (J_c) of superconducting $\text{GdBa}_2\text{Cu}_3\text{O}_7$.

Sintering (°C)	$T_{C(R=0)}$	$T_{C\text{ (onset)}}$	Impurities %	Relative Density %	Porosity %	J_c (A/cm ²)
920	95	97	3.8	80.3	19.7	4.4±0.1
930	94	97	0.9	83.2	16.8	6.4±0.4
940	94	96	1.8	89.9	10.2	7.9±0.7
950	95	97	2.2	95.3	4.7	12.9±0.9

4. Conclusions

$\text{GdBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconducting ceramics were synthesized from nanoscale metal-oxalate precursors via co-precipitation, followed by sintering at temperatures ranging from 920 °C to 950 °C. Elevated sintering temperatures resulted in an increase of the orthorhombic Gd123 phase fraction from 92% to 99.8%. Electrical measurements demonstrated metallic characteristics, with (T_c , $R=0$) consistently observed between 94 and 95 K.

Both XRD and SEM analyses show that raising the sintering temperature improves the critical current density. According to XRD patterns, small amounts of secondary phases are present, which can serve as efficient flux-pinning centers. Meanwhile, SEM images demonstrate that higher temperatures progressively decrease the gaps and voids between superconducting grains. This reduction in insulating cavities strengthens intergranular coupling and increases the material's relative density, resulting in better current transport efficiency.

Overall, these findings demonstrate that careful optimization of sintering temperature can significantly enhance the microstructure and transport properties of Gd123 ceramics, offering valuable guidance for their development in large-scale electrical and magnetic applications.

Author Contributions: Conceptual: Ahmed Al-Mobydeen and Imad Hamadneh; methodology: Mohammed M Alawamleh.; software: Imad Hamadneh and Jamal Rahhal; validation: Yousef Al-Dalahmeh, Sondos Shamah and Jamal Rahhal; formal analysis: Wala'a Al-Tarawneh and Sondos Shamah; investigation: Mohammed M Alawamleh and Ahmed Al-Mobydeen.; data curation: Imad Hamadneh.; writing—original draft preparation: Imad Hamadneh.; writing—review and editing: Iessa Sabbe Moosa and Ehab AlShamaileh.; visualization: Iessa Sabbe Moosa, Ehab AlShamaileh and, Mike Haddad; supervision, Imad Hamadneh.; project administration: Imad Hamadneh.; funding acquisition: Imad Hamadneh. All authors have read and agreed to the published version of the manuscript.

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