



A Modified Thermal Decomposition Approach for The Synthesis of Phase-Pure $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ Superconductor

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Abstract: High temperature superconductor of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (Y-123) has been synthesised using the modified thermal decomposition method (MTD). In this work, a pure Y-123 sample is produced using an acetate-based material as a precursor and sintered at 980 °C. The study on the phase formation, superconducting properties and microstructural characteristics of pure Y-123 was investigated using X-ray diffraction (XRD), four-point probe (4PP) and scanning electron microscopy (SEM) respectively. XRD analysis confirmed the formation of a single-phase orthorhombic Y-123 crystal structure with an orthorhombicity of 0.008, indicating high phase purity without detectable impurity phases. Electrical measurements revealed a sharp superconducting transition with a critical onset temperature, $T_{c\text{-onset}}$ of 97.3. SEM observations showed plate-like grains with an average grain size of 2.25 μm and good grain connectivity. These results demonstrate that the modified thermal decomposition technique is an effective approach for producing phase-pure YBCO superconductors with excellent structural and superconducting properties, highlighting its potential for advanced superconducting applications.

Keywords: Superconductor; Thermal Decomposition Method; YBCO

Introduction

Superconductors are a special class of materials that, when cooled below a distinctive critical temperature (T_c), have their resistivity suddenly become zero (Rizwan et al., 2022). Materials exhibit zero direct-current electrical resistance and the Meissner effect, which allows them to expel magnetic fields from their interiors (Sorongane, 2023). These characteristics set superconductors apart from ordinary conductors in fundamental ways, making them materials of significant scientific and technological value. Among copper-oxide based materials, Yttrium Barium Copper Oxide ($\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$), commonly known as YBCO has become one of the most extensively studied high-temperature

superconductors due to its relatively high critical temperature (~ 92 K) and good superconducting performance. $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ is one of the high-temperature superconductors that has garnered significant interest due to its chemical stability, practicality, and relatively high critical temperature above the boiling point of liquid nitrogen (77 K) (Düzgün, 2025). Because of this ability, superconductors attracted considerable interest in technological applications such as power transmission, magnetic energy storage (Arsénio Costa & Morais, 2024), medical imaging systems (Xu et al., 2025) and transportation technologies (Deng et al., 2021).

The superconducting properties of YBCO are highly dependent on its phase purity, oxygen

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stoichiometry, and microstructural characteristics, including grain size and grain boundary connectivity. Therefore, the synthesis technique plays a crucial role in determining the final quality of the material. Various preparation methods have been developed to synthesize YBCO superconductors, including the conventional solid-state reaction method (Onar & Yakinci, 2015), sol-gel processing (Abdul Hussein et al., 2023), thermal treatment method (Dzul-Kifli et al., 2025) and thermal decomposition techniques (Sah et al., 2024). Although the solid-state method is widely used for its simplicity, it often requires high processing temperatures and prolonged heat treatments, which can lead to poor homogeneity and the formation of unwanted impurity phases. Other than that, solid-state processes frequently go through a number of nonequilibrium intermediates before reaching the equilibrium phase, according to recent in site characterization investigations. Trial-and-error methods are used to optimize ceramic synthesis because this intricate phase development is currently poorly understood. It is difficult to comprehend these reaction pathways (Miura et al., 2021). Alternative chemical synthesis routes have been introduced to overcome these limitations and to provide better control over particle size, chemical homogeneity, and phase formation.

Among these approaches, the modified thermal decomposition method (MTD) has gained attention, as it offers the potential to improve superconducting characteristics and to better regulate material properties. In particular, careful control of thermal processing steps such as calcination, sintering, and oxygenation is required to obtain the desired orthorhombic Y-123 structure and optimal superconducting performance. Modified thermal decomposition procedures optimize calcination temperature, heating rate, dwell time, and oxygenation conditions to further enhance superconducting characteristics. These changes are intended to increase uniform oxygen diffusion, enhance grain connection, and inhibit the formation of secondary phases.

In this study, pure YBCO superconductors were synthesized using a Modified Thermal Decomposition (MTD) method. This technique involves controlled thermal processing of metal acetate precursors, including yttrium (III) acetate tetrahydrate, barium acetate, and copper (II) acetate monohydrate, followed by precisely controlled heating stages which are pre-calcination, calcination and sintering. The aim of this work is to achieve superior purity, microstructure and density compared to traditional methods especially for complex ceramics like Y-123 superconductors. The effectiveness of the MTD method in producing pure YBCO superconductors with good structural, microstructural, and electrical properties were

characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and four-point probe electrical measurements to evaluate their crystallographic structure, morphology, elemental composition, and superconducting behavior.

Method

The material used in this study was selected based on its suitability for producing phase-pure YBCO superconductors via the modified thermal decomposition method. Yttrium (III) acetate tetrahydrate, $(Y(OOCCH_2)_2 \cdot 4H_2O, 99.9\%)$, barium acetate $(Ba(CH_3COO)_2)$ and copper (II) acetate monohydrate, ACS $(Cu(CH_3COO)_2 \cdot H_2O, 98.0-102.0\%)$, were weighed with ratio 1:2:3 stoichiometric amounts. These metal precursors were from Thermo Scientific while for barium acetate from R&M Chemicals.

The materials were then stirred manually using a glass rod for 30 minutes to encourage close molecular interaction between the precursors. The mixture is then pre-calcined at 600 °C for 30 minutes in the box furnace to remove volatile components. This stage promotes more consistent phase development during calcination. The powder was ground for 1 hour before being calcined for 18 hours at 910 °C. Later, the sample is pressed into a pellet (13 mm in diameter and 2 mm thick) and compressed to 5 tons using a hydraulic press. The bulk sample was then sintered for 24 hours at 980 °C and slowly cooled down to 600 °C for the annealing process. The sintering process is crucial to improve the intergranular connection and densification of the samples.

Result and Discussion

X-Ray Diffraction (XRD) Analysis

The X-ray diffraction pattern for pure Y-123 synthesised using the MTD method. Based on the graph in Figure 1, no detectable secondary phases, such as Y_2BaCuO_5 (Y-211) or $BaCuO_2$, are observed, indicating that the MTD synthesis process successfully inhibited the formation of impurity phases. This demonstrates that the applied heat-treatment profile and precursor homogeneity were sufficient to promote the full phase development of YBCO. Meticulous thermal control produced a dominant Y-123 phase with few secondary phases (Sah et al., 2024). In addition, the clear splitting of characteristic peaks, particularly between peak (020) and (200) reflections, confirms the orthorhombic crystal structure that is essential for superconductivity in YBCO. The prominent diffraction peak that appears at about $2\theta \approx 32^\circ$ corresponds to the (103) reflection, a characteristic peak of the orthorhombic YBCO phase,

and the sample is confirmed to have crystallised in the superconducting orthorhombic phase. The maximum peak of the major phase Y123 was observed at $2\theta = 32.84^\circ$ for the (110) planes, indicating that all samples were consistently in the orthorhombic phase (Kamarudin et al., 2022).

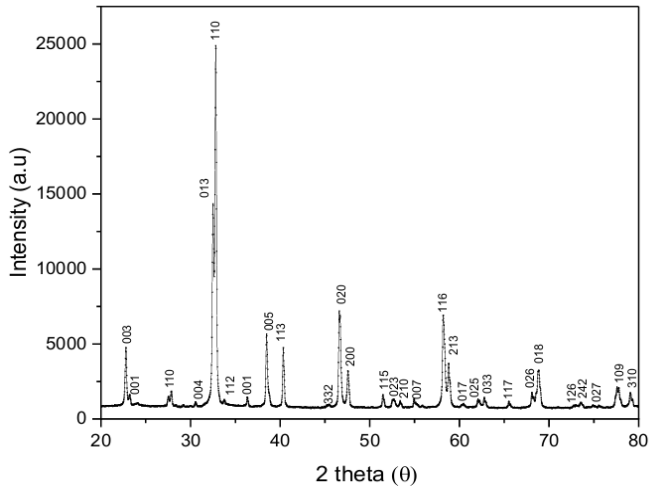


Figure 1. XRD pattern for pure Y-123 sample synthesized using modified thermal decomposition method.

Table 1 shows the information of the crystallite size, lattice parameter, lattice strain and orthorhombicity of pure YBCO. Pure YBCO, showing $a = 3.818 \text{ \AA}$, $b = 3.883 \text{ \AA}$, and $c = 11.676 \text{ \AA}$, which are in good agreement with crystallite values reported for orthorhombic $\text{YBa}_2\text{Cu}_3\text{O}_7$ (Akhtar, 2011). The orthorhombicity value is

calculated using the formula $[(b - a)/(b + a)]$, where a is lattice constant a and b is lattice constant b (Dihom et al., 2017). The orthorhombic value is 0.008. This result verifies the existence of an oxygen-ordered orthorhombic phase and shows a distinct departure from the tetragonal structure. Higher orthorhombicity is associated with better oxygen ordering along the Cu–O chains, which is necessary for effective charge transport and improved superconducting performance (Arebat, Kechik, Baqiah, et al., 2025).

The calculated lattice strain of 0.23% indicates minimal internal deformation of the crystal lattice. Good structural stability and a lower concentration of defects are 41 indicated by low lattice strain, which is advantageous for preserving superconducting pathways. The average crystallite size of approximately 733 \AA (73.3 nm). The average crystallite size can be calculated from (103) peak by using the Scherrer equation 1.

$$D_{hkl} = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

where k is shape factor which usually is taken as 0.9, λ is the wavelength of XRD $\text{CuK}\alpha$ radiation source (nm), β is the highest intensity of the main peak and θ is the bragg angle (Monshi et al., 2012). Larger crystallite sizes are beneficial because they improve superconducting current transport and lessen grain boundary scattering (Sah et al., 2024).

Table 1. crystallite size, lattice parameter, lattice strain and orthorhombicity of pure Y-123

Sample	Crystallite size		Lattice Parameter		Lattice strain (%)	Orthorhombicity
	(\AA)	$a(\text{\AA})$	$b(\text{\AA})$	$c(\text{\AA})$		
Pure YBCO	733	3.82	3.88	11.67	0.23	0.008

Electrical Resistance Measurement by Four-Point Probe Technique

The resistance as a function of temperature for the pure YBCO sample as shown in Figure 2. The sample displays metallic behavior at high temperatures over 100 K. Good charge carrier mobility within the grains is indicated by this metallic trend, which is typical of YBCO in its normal (non-superconducting) state (Arebat, Kechik, Hong, et al., 2025).

Table 2. Table of resistance against temperature of pure Y-123

Sample	$T_{c\text{-Onset}}$ (K)	$T_{c\text{-Offset}}$ (K)	ΔT_c (K)
Pure YBCO	97.3	93.3	4.0

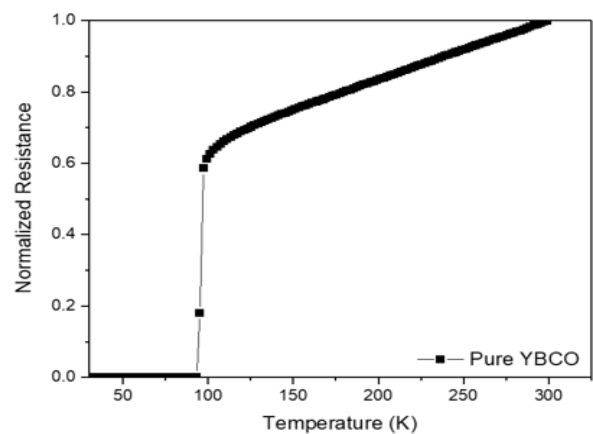


Figure 2. Normalized resistance as a function of temperature for the pure Y-123 sample.

Based on Table 2, the onset critical temperature, T_{c_onset} is measured at 97.3 K while offset critical temperature, T_{c_offset} is reached at 93.3 K and yielding a transition width ΔT_c of 4 K. The narrow ΔT_c value indicates the degree of homogeneity, disorder and inter-grain coupling in the superconducting material. Minimal non-ideal effects, such as intergranular resistivity, structural disorder and compositional inhomogeneity, are implied by a tiny ΔT_c . Thus, this study of ΔT_c value of 4 K indicates minimal weak-link behavior and strong grain connection between superconducting grains (Mazaheri et al., 2015). While broader transitions are frequently linked to impurity phases or the coexistence of several phases, which reduce intergranular coupling. A high value in T_c may be related to a rise in the amount of oxygen in the CuO chain, which is produced by a decrease in the per CuO₂ plane, that also be related to the sample's homogeneity (Sah et al., 2024).

Microstructure analysis

The image from the SEM under 3000x magnification as shown in Figure 3. The plate like grains (Hasan et al., 2021) describe the microstructures of the characteristic of YBa₂Cu₃O₇ superconductors. These plate-form grains overlap and interlock, forming a network of interconnected grains across the surface. There are small holes between neighbouring grains in the surface morphology, indicating modest porosity. Nonetheless, the majority of the grains are in close proximity to one another, which indicates comparatively good densification during sintering, and the pores are not prominent. YBCO grains can grow in either faceted and porous morphologies or smooth and dense forms depending on growth circumstances and crystallographic orientation. Both faceted grain edges and comparatively smooth plate surfaces are seen in the current investigation with grain-to-grain changes in grain width (Pahlke et al., 2016)

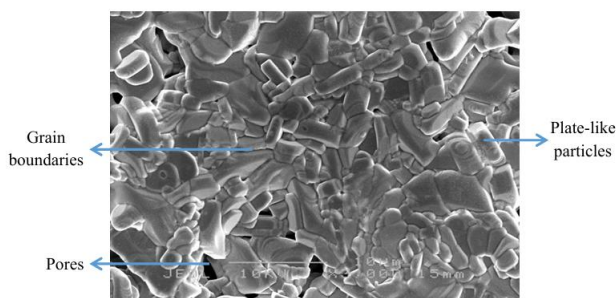


Figure 3. SEM image of YBCO under 3000x magnification for pure Y-123 sample.

Table 3 summarizes the average grain size is roughly 2.25 μm , suggesting consistent grain growth

across the sample. The grain size may be rises with addition such as cobalt oxide addition (Yunus et al., 2018). Variations in atomic orientation between nearby grains throughout the development phase cause grain borders to emerge. The interconnected plate-like grains and comparatively low pore density in the current sample imply that the grain borders are well-connected, reducing weak-link behaviours (Ezzatpour et al., 2018),

Table 3. Average grain size of pure Y-123

Sample	Average grain size (μm)
Pure YBCO	2.25

Energy Dispersive X-Ray (EDX) Analysis

Energy-dispersive X-ray analysis is used to determine the elemental composition of a sample. Figure 5 show the EDX spectra and random spot grain selection on the SEM image to guarantee representative elemental information of the bulk material. There are distinct, well-defined peaks that clearly correlate to the elements yttrium (Y), barium (Ba), copper (Cu), and oxygen (O). The absence of contamination and the effective synthesis of pure YBCO were confirmed by the lack of additional peaks associated with impurities or secondary elements.

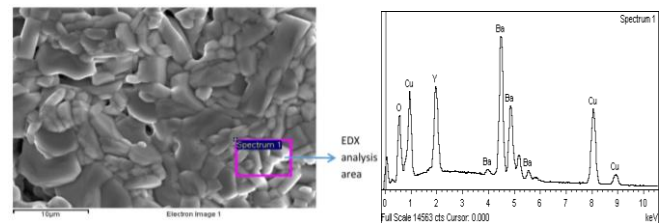


Figure 5. EDX spectra for the Y-123 sample

Table 4. Atomic percentage of element Pure YBCO

Atomic %				Atomic ratio Y: Ba:Cu:O
Y	Ba	Cu	O	1: 1.7: 2.3: 5.2
9.75	16.80	22.27	51.18	

Table 4 shows the quantitative elemental analysis derived from the EDX spectrum. Y, Ba, Cu, and O were found to have atomic percentages of 9.75%, 16.80%, 22.27%, and 51.18%, respectively. The computed atomic ratio of Y based on these values of Y:Ba:Cu:O was roughly 1: 1.7: 2.3: 5.2. The EDX results verify that all of the expected elements Y, Ba, Cu and O for the synthesis of YBa₂Cu₃O₇. The ideal stoichiometric ratio of 1:2:3, which is anticipated for YBCO superconductors, is similar to the measured cation ratio Y:Ba:Cu. EDX atomic ratios of Y:Ba:Cu ratios for thermal-synthesised YBCO samples were typically around the optimal 1:2:3 ratio, especially when random spot analysis was used. In addition, emphasized that during the EDX

measurement, may result in the Y:Ba:Cu ratio because of the random selection of analytical points particularly in polycrystalline materials with varied grain orientation and surface topography (Dzul-Kifli et al., 2022)

Conclusion

The pure $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductor material was successfully synthesized by using a Modified Thermal Decomposition method. The results from the XRD analysis confirmed that the material was of high purity, had an orthorhombic distortion, and was therefore a good superconductor material. The electrical properties of the material, which were determined by using the four-point probe method, showed that it was an excellent superconductor material with a high onset critical temperature of 97.3 K, a narrow transition width of 4.0 K, and was metallic above T_c . The microstructural analysis of the material which was done by using SEM showed that it had plate-like grains with an average grain size of 2.25 μm , which were well connected, thereby reducing weak-link effects. The EDX analysis of the material confirmed that it had an elemental composition close to the ideal stoichiometry of Y:Ba:Cu. In conclusion, the MTD method proved to be an effective, cost-efficient, and viable technique for manufacturing high-quality YBCO superconductors with properties that can support sophisticated applications such as maglev trains, MRI magnets, and energy-efficient power transmission.

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Author Contributions

Conceptualization, M.M.A.K.; methodology, N.A.C.D.-K., Y.S.H and N.H.M.H; formal analysis, N.Z.Z.; investigation, M.M.A.K.; writing—original draft preparation, N.A.C.D.-K.; writing—review and editing, M.M.A.K. and M.K.S.; supervision, M.M.A.K., S.K.C., A.H.S. and K.P.L.; project administration, M.M.A.K.; funding acquisition, A.D. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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