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Comparative Study of Y123 Superconductors Synthesized Under Open Air and Oxygen Flow Conditions

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Abstract: $YBa_2Cu_3O_{7-\delta}$ (Y123) superconductors is a widely studied high-temperature superconductor due to its high critical temperature, T_c and strong flux pinning properties. In this study, Y123 samples were synthesized via a thermal treatment method under two sintering conditions which were open air and oxygen flow. Structural, microstructural, and superconducting properties were evaluated using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and AC susceptibility measurements. XRD analysis revealed that all Y123 samples exhibited predominantly Y123 phase with orthorhombic structure, with minor secondary phases. The Y123 sample prepared in an open-air condition exhibited larger grain size (0.698 µm), lower porosity, and fewer impurities compared to sample prepared in the oxygen flow condition. AC susceptibility showed a higher $T_{c\text{-onset}}$ exhibited at 92.1 K in the open-air sample, indicating better grain connectivity. These results suggest that open-air sintering offers a simpler, cost-effective route for enhancing Y123 superconductor performance.

Keywords: Critical temperature; Thermal treatment; Microstructure; $T_{c\text{-onset}}$; Y123

Introduction

High-temperature superconductors (HTS) have attracted significant attention due to their ability to exhibit zero electrical resistance and perfect diamagnetism above the boiling point of liquid nitrogen (77 K) (Sheahen, 1994). Yttrium barium copper oxide or $YBa_2Cu_3O_{7-\delta}$ (Y123) is one of the most studied HTS compounds since its discovery in 1987 showin its high critical temperature, T_c of 92 K with their excellent current-carrying capability, and chemical stability (Wu et al., 1987). These properties make Y123 a strong candidate for various technological applications, including power transmission cables, fault current limiters, magnetic levitation systems, and superconducting magnets (Liu et al., 2018; Masuda et al., 2005; Mukoyama et al., 2007; Yildizer et al., 2016).

The synthesis of Y123 superconductors can be carried out via several methods, such as sol-gel, co-precipitation, and modified thermal decompositions (Abdullah et al., 2023; Dadras et al., 2017; Kamarudin et al., 2022; Sah et al., 2024) However, the thermal treatement method remains the most widely used due to its simplicity, scalability, and cost-effectiveness (Dihom et al., 2017). This technique typically involves multiple calcination and sintering steps to promote phase formation and grain growth. The final microstructure and superconducting properties of Y123 are highly sensitive to processing conditions, particularly the sintering atmosphere, temperature, and oxygenation. Also, thermal treatment method under different oxygen environments significantly influences oxygen stoichiometry, lattice structure, grain connectivity, and the formation of secondary phases, which in turn affect the superconducting performance (Dihom, Shaari, Baqiah, Al-Hada, Kean, et al., 2017; Hannachi et al., 2022; Konstantinov et al., 1996).

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Despite extensive research, optimizing the processing parameters especially the sintering conditions for producing a good Y123 superconductors remains a challenge. Excess oxygen flow during sintering can lead to the formation of impurity phases like $BaCO_3$, while insufficient oxygenation may disrupt the orthorhombic structure critical to superconductivity. Therefore, identifying the optimal sintering environment is essential to balance phase purity, microstructure, and superconducting characteristics. Therefore, the aim of this study are to synthesize Y123 samples via the thermal treatement method under two different sintering conditions which was open air and oxygen flow. A systematic study on structural, microstructural, compositional, and superconducting properties were conducted. Y123 sample sintered in open air exhibited improved grain morphology, reduced porosity, fewer impurities, and a higher $T_{c\text{-onset}}$ (92.1 K) compared to the oxygen-sintered sample (91.3 K). These findings suggest that open-air sintering may offer a more effective and scalable route for producing high-quality Y123 superconductors.

Method

Y123 samples were synthesized using the metal nitrates such as $Y(NO_3)_3.6H_2O$, $Ba(NO_3)_2$ and $CuN_2O_6.2.5H_2O$ by following the stoichiometric equations of 1:2:3 and dissolved in 300 mL of deionized water with 2 % aqueous solution of polyvinyl pyrrolidone (PVP), which acts as a capping agent. The mixed solution was stirred at 80 °C with 850 rpm for 2 hours to ensure homogenous mixing of the metal precursors and PVP (Zahari et al., 2017). The slight blue solution obtained was dried in the oven for 24 hours at 110 °C to remove the water. Then, the dark green gel-like samples obtained from the drying process were ground by using the mortar and pestle until it formed a fine powder. Fine powders undergo a two-step calcination process for 12 hours at 600 °C and 24 hours at 910 °C, respectively, in the box furnace with intermediate grinding and cooled in air with a cooling rate of 1 °C per minute. Then, the fine powdered samples were reground and pressed into a 1-gram pellet at 5.5 tons of pressure before sintered. The sintering temperature at 980 °C was conducted for Y123 samples with duration of 24 hours and slowly cooled to room temperature for 1 °C per minute cooling rate. In this study, two sets of Y123 samples were sintered in two different atmospheres, which were in an open-air and oxygen-atmospheric environment, following the heating pattern in Figure 1.

All Y123 samples obtained were characterized using X-ray diffraction (PW 3040/60 MPD X'Pert Pro Panalytical Philps DY 1861 X-ray diffractometer with CuKα radiation source (λ=1.5406Å) operated at 40 mA and 40 kV in angle 2θ range 20° to 80° with scanning step size of 0.03° to study the phase formation and crystal structure of the samples. Then, the surface morphology of the Y123 samples was observed through a scanning electron microscope (SEM-LEO 1455 VPSEM) equipped with an energy dispersive X-ray spectrometer (EDX). Lastly, the superconducting magnetic properties of the transition temperature, $T_{c\text{-onset}}$, using an AC-susceptometer (ACS), CyroBIND (Cryogenic Balanced Inductive Detector), SR830 lock-in amplifier at the frequency of 219 Hz with a 1 Oe magnetic field.

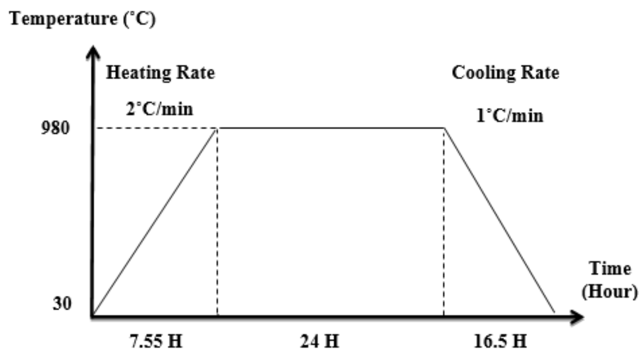

Figure 1. Sintering process at 980˚C with heating and cooling curve for 24 hours process.

Results and Discussions

X-ray Diffraction Analysis

In this study, the diffraction patterns for both samples sintered in open air and under flowing oxygen were analyzed using X'Pert HighScore software. As shown in Figure 2, it was observed that the phase indexed to the orthorhombic phase of Y123, indicating successful formation of the superconducting phase in both atmospheric conditions. The dominant diffraction peak for both samples corresponds to the [1 0 3] reflection, characteristic of the Y123 phase, consistent with previous literature reports (Ramli et al., 2016; Sahoo et al., 2019). The intensity of the [103] peak is higher for the sample sintered in open air, suggesting a variation in texture or crystallinity depending on the sintering atmosphere. Despite the dominance of Y123 peaks, minor reflections corresponding to secondary phases such as Y_2BaCuO_5 (Y211) and $YBa_2Cu_4O_8$ (Y124) were also detected and are labelled in Figure 2 which were commonly reported in YBCO systems and typically arise due to partial decomposition or incomplete reaction during the sintering or annealing process (Wang et al., 2025).

The formation of the Y211 phase is often attributed to thermodynamic instability in the Y123 matrix under certain oxygen partial pressures. Previous reported study mentioned that the decomposition of Y123 into Y211 and other stable oxide phases may occur during oxygen annealing, particularly when the local oxygen content is not adequately maintained. The presence of

Y211 can also serve as an indicator of defects or inhomogeneities in the microstructure, potentially affecting the flux pinning behaviour and critical current density of the final material (Diko et al., 2008; Wang et al., 2025). Also, it was observed that increase in intensity at 2θ = 20.29 ° and 23.36 ° was observed in the open-air sintered sample, which could be associated with enhanced crystallinity or slight changes in oxygen stoichiometry. However, unmatched peaks also appear in the diffractograms, which may be attributed to either residual impurities or background noise, as they do not correspond to known YBCO related phases. Nevertheless, these findings underscore the significant influence of the sintering atmosphere on the phase composition and structural integrity of YBCO materials. Control over the thermal treatment environment is therefore critical to optimize the superconducting properties of the resulting ceramics.

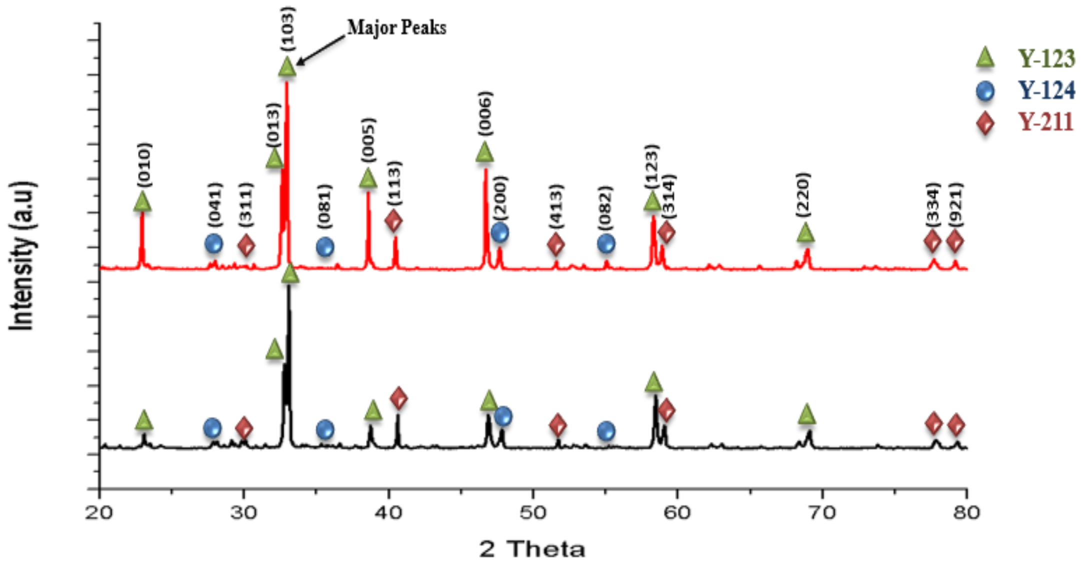

Figure 2. XRD pattern of pure YBCO-123 that sintered at different condition which is oxygen flow (black line) and open air (red line). There are Y-211 phase labelled with red diamond symbol and Y-124 phase that labelled as blue sphere symbol in the graph

The lattice parameters of *a,b, c* and volume of unit cell of Y123 at different conditions were tabulated in Table 1. It was observed that the Y123 samples sintered in open air exhibit slightly reduced values for the *a*-axis and *c*-axis while the *b*-axis parameter increases relative to the samples sintered under flowing oxygen. This behavior is correlated with previous study observed that oxygen deficiency disrupts the Cu-O chains, contracting the *a*-axis and *c*-axis and expanding the *b*-axis (Howe, 2014). In addition, it was observed that the cause of lattice distortions were influenced the total unit cell volume, which is found to be 173.65 Å^3 for the oxygen-sintered sample and 173.548 Å^3 for the open-air sample.

Table 1. The lattice constants for sample YBCO-123 sintered with oxygen flow and open-air atmosphere.

Sample	Lattice parameter			Unit cell
	a (Å)	*b* (Å)	*c* (Å)	volume (Å^3)
Oxygen flow	3.823	3.887	11.686	173.653
Open air	3.821	3.889	11.679	173.548

This minor reduction in cell volume under oxygen-deficient conditions reflects the structural strain caused by reduced oxygen content and is consistent with the Rietveld refinement results reported by Benzi et al. (Benzi et al., 2004). The changes in lattice parameters and unit cell volume are not only indicative of variations in oxygen incorporation but are also critical for understanding the superconducting properties of Y123. Thus, the enhancment during sintering leads to more uniform and expansive lattice structures, which are essential for optimal charge carrier mobility and superconducting performance.

SEM analysis

To further investigate the effect of sintering conditions on the microstructure of Y123, the observations by SEM was employed at magnifications of 1500x. The representative micrographs of both surface and cross-sectional areas are shown in **Figures 3.** SEM images revealed that the Y123 grains exhibit irregular shapes and loose boundary connectivity in both samples. However, it was noticeable that the sample sintered under flowing oxygen exhibited a higher degree of porosity and smaller, more fragmented grains, compared to the open-air sintered sample shows denser packing with larger grains and fewer voids. This microstructural variation were observed by Konstantinov et al., who reported that higher oxygen partial pressure can suppress grain growth and lead to finer microstructures in YBCO ceramics (Konstantinov et al., 1996).

The average grain size, as shown in Table 2 was measured to be 0.698 µm for the open-air sample and 0.566 µm for the sample sintered in oxygen flow. The coarserned of the grains in the open-air sample suggested to the formation of large grains due to reduced oxygen diffusion during sintering which demonstrated that lower oxygen pressures promote better grain connectivity in Y123 (Pathak et al., 2001). Moreover, previous study also reported an average grain size which closely aligns with the open-air sample result obtained from this study (Dihom, Shaari, Baqiah, Al-hada, Abidin, et al., 2017). The comparatively smaller grain size in the oxygen-flow sample may also be attributed to the presence of secondary phases or impurities such as $BaCO_3$ which can inhibits to the grain growth (Konstantinov et al., 1996).

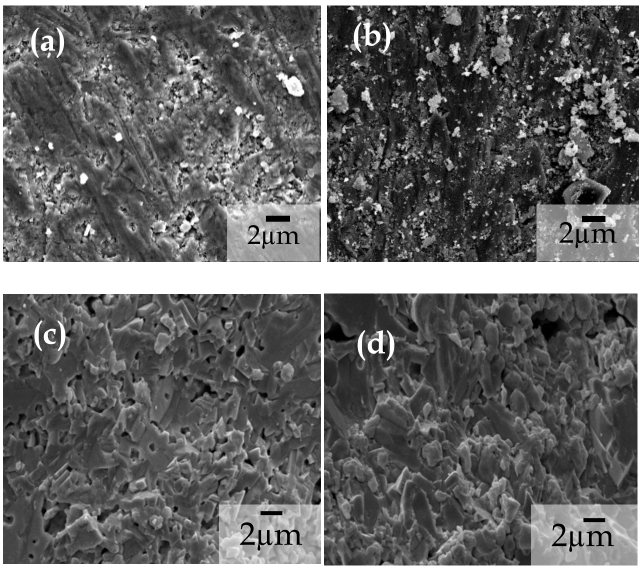

Figure 3. SEM images of YBCO-123 surface morphology and cross-sectional microstructure at different sintering condition which (a),(c) oxygen flows and (b),(d) open-air at 1500X magnifications.

Also, it was observed that the formation of higher porosity was observed in open-air sintered sample with 0.323 µm, while the oxygen-sintered counterpart exhibits larger pores averaging 0.438 µm. Lower porosity is typically beneficial for superconducting transport, as it enhances grain-to-grain contact and facilitates better current percolation (Benzi et al., 2004). Moreover, finer and well-distributed pores can act as effective flux pinning centers during superconducting operation, thereby improving the J_c performances (Kamarudin et al., 2025). These findings shows the influence of the sintering atmosphere on optimising the microstructure and, ultimately, the functional properties of YBCO superconductors were necessary.

EDX analysis was conducted for Y123 samples sintered under different atmospheric conditions as shown in Figure 3. It was revealed that both Y123 samples reflected the yttrium (Y), barium (Ba), copper (Cu), oxygen (O), and carbon (C) elements with expected composition of Y: Ba: Cu that was close to the ratio of 1: 2: 3 as tabulated in Table 2. However, it was notably observed that carbon (C) was detected only in the oxygen-sintered sample, suggesting the formation of barium carbonate ($BaCO_3$), an impurity often produced during high-temperature synthesis in oxygen-rich environments, as supported by earlier studies (Konstantinov et al., 1996). The atomic ratios show deviations from the ideal stoichiometry, with the oxygen-sintered sample exhibiting a higher oxygen content and more pronounced deviation in the Ba and Cu ratios. In contrast, the open-air sintered sample displays a more balanced composition with no detectable carbon, indicating fewer carbonate impurities and potentially better phase purity. These findings highlight that excessive oxygen flow during sintering may facilitate impurity formation and disturb the stoichiometric balance of Y123, whereas open-air sintering offers a more controlled environment for maintaining structural and compositional integrity (Cava et al., 1990).

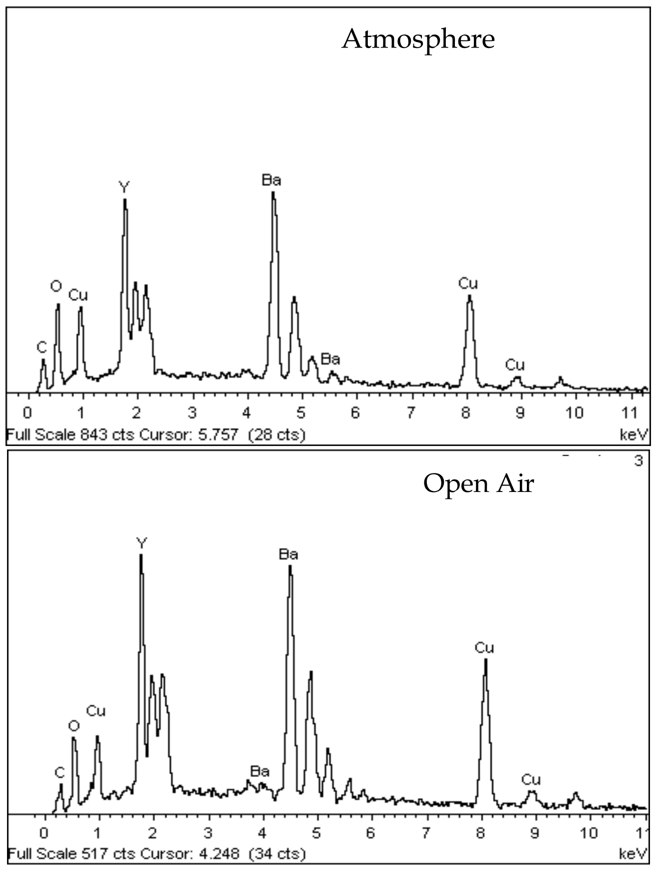

Figure 4. The EDX spectra line for YBCO-123 sintered at different condition which is oxygen flow and open-air.

Table 2. The ratio of $YBa_2Cu_3O_{(7-\delta)}$ (YBCO-123) atomic percentage at different sintering condition

Condition	Y (%)	Ba (%)	Cu (%)	O (%)	Average Y-123 grain sizes (µm)
Oxygen Flow	2.93	7.14	9.44	36.54	0.566
Open Air	4.01	8.81	13.01	30.61	0.698

AC-Susceptibility (ACS)

The superconducting transition temperature of $T_{c\text{-}onset}$ was determined through magnetization measurements using AC susceptibility analysis. Figure 5 displays the temperature-dependent susceptibility curves of the $T_{c\text{-}onset}$ corresponds to the intragranular transition, where individual grains begin to exhibit superconductivity. From the graph, the $T_{c\text{-}onset}$ for Y123 samples as observed at 92.1 K for the sample sintered in open air, while the sample sintered under oxygen flow exhibited a slightly lower $T_{c\text{-}onset}$ at 91.3 K. These results reveal that the open-air sample not only initiates superconductivity at a higher temperature but also exhibits a broader transition width (ΔT_c = 12.6 K) compared to the narrower transition range observed in the oxygen-sintered sample (ΔT_c = 8.1 K). A broader ΔT_c is typically associated with more structural or compositional inhomogeneity (Veneva et al., 1998).

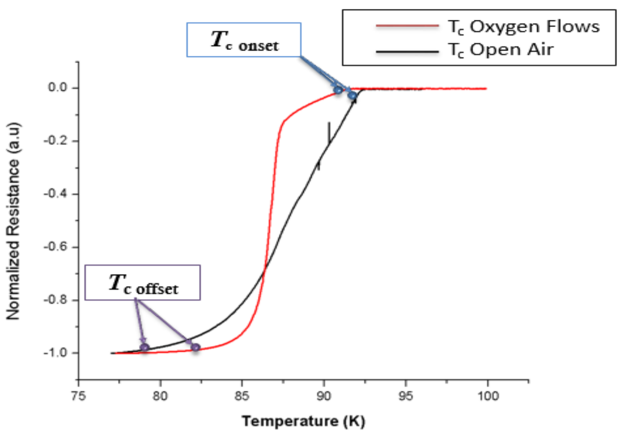

Figure 5. The ACS results that show transition temperature of the YBCO-123 that sintered at different condition which is oxygen flows and open air.

However, the higher $T_{c\text{-}onset}$ suggests enhanced grain connectivity in the open-air sample. This finding is in agreement with earlier work by Pathak et al., who reported that reduced oxygen partial pressure during sintering enhances grain linkage, leading to improved superconducting transition characteristics (Pathak et al., 2001). In the present study, an oxygen flow rate of 35 cc/min was used during sintering, which may have contributed to over-oxygenation and the formation of secondary phases or microstructural defects, slightly reducing T_c in that sample. Therefore, the results revealed that sintering atmosphere plays a crucial role in tuning the superconducting transition behavior of Y123 ceramics.

Conclusion

Y123 superconductors were successfully synthesized via thermal treatment under open-air and oxygen flow conditions. XRD analysis confirmed the formation of the orthorhombic Y123 phase in both samples, with minor secondary phases of Y211 and Y123. Lattice parameter variations suggest better oxygen stability in the open-air sample. SEM and EDX analysis revealed that open-air sintering promotes larger grain size, lower porosity, and fewer carbonate-related impurities compared to oxygen flow. AC susceptibility results showed a higher $T_{c\text{-}onset}$ (92.1 K) for the open-air sample, indicating enhanced grain connectivity. Overall, open-air sintering yields superior structural and superconducting properties, making it a more favorable and cost-effective route for Y123 fabrication.

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Author Contributions
Conceptualization, M.M.A.K, A.N.K and M.A.K..; methodology, M.A.K.; software, A.N.K.; validation, M.M.A.K., C.S.K. L.K.P, A.D. and A.H.S.; formal analysis, M.A.K., A.N.K. and M.M.A.K.; investigation, M.A.K. and A.N.K.; resources, M.M.A.K.; data curation, M.A.K. and A.N.K.; writing—original draft preparation, M.A.K. and M.M.A.K.; writing—review and editing, A.N.K., M.M.A.K., A.D.; visualization, M.K.A.K. and M.K.S.; supervision, C.S.K., L.K.P. and A.H.S.; project administration, M.M.A.K.; funding acquisition, M.M.A.K and 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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