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Influence of Carbon Nanotubes in Improving the Superconducting and Structural Properties of Bulk Bi-2212 Synthesis by Thermal Treatment Method

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Abstract: Bi-2212 superconductor has garnered significant interest in recent years due to its potential applications in the development of superconducting wires and tapes. The effect of introducing carbon nanotubes (CNT) into the Bi-2212 system was investigated in terms of superconducting and structural properties. The results of this study indicated that the addition of CNTs had a notable effect on the phase formation of Bi-2212, with a substantial increase from 86.8% to 97.4% for the sample with a weight percentage of 0.8 wt.%. This can be attributed to the improved particle orientation brought about by the introduction of CNTs. The microstructure analysis displayed randomly distributed grains of irregular shapes, with a reduced average grain size of 1.018 µm upon the addition of 0.4 wt.% CNTs. Additionally, the inclusion of CNTs led to an increase in the T_c value, with the maximum $T_{c\text{-onset}}$ recorded at 79 K for the sample containing 0.6 wt.% CNTs. In summary CNT has enhanced the structural and the superconducting properties of the Bi-2212 synthesised with thermal treatment method.

Keywords: Bi-2212; Thermal treatment; Carbon nanotubes; BSCCO

Introduction

Bismuth strontium calcium copper oxide, commonly referred to as BSCCO, represents a significant breakthrough in the field of high-temperature superconductivity as it does not contain any rare earth elements. As a result, extensive research efforts have been dedicated to studying the superconducting properties of BSCCO, making it an ideal material for the production of superconducting wires. The composition of BSCCO, denoted by the formula $Bi_2Sr_2Ca_nCu_{n+1}O_{2n+6+\delta}$ (n = 0, 1, and 2), comprises of three distinct phases. Specifically, these phases are identified as Bi-2201 (n = 0), Bi-2212 (n = 1), and Bi-2223 (n = 3). Notably, the critical temperature (T_c) for each of these phases differs, with Bi-2201 exhibiting a T_c of 10 K, Bi-2212 a T_c of 85 K, and Bi-2223 a T_c of 110 K. In this study, the Bi-2212 phase was chosen as it is economically feasible, environmentally friendly, and thermodynamically stable (Özkurt & Aytekin, 2018).

Numerous techniques have been utilized for synthesizing BSCCO, employing either dry or wet methods, including solid-state (Özkurt & Aytekin, 2018; Arlina, Shaari, Chen, Mohamed, Mohd Kamal, & Kechik, 2019), co-precipitation (Hamadneh, Agil, Yahya, & Halim, 2007; Sotelo, Rasekh, Madre & Diez, 2011), sol-

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gel (Fallah-Arani, Baghshahi, Sedghi, Stornaiuol, Tafuri, Massarotti, Riahi-Noori, 2018; Sharma, Kumar & Awana, 2013), and thermal treatment (Dzul-kifli, Kechik, Baqiah, & Shaari, 2022; Kamarudin, Kechik, Abdullah & Baqiah, 2022; Barood, Kechik, Tee & Kien, 2023). In particular, Dihom, Shaari, Baqiah, & Al-hada (2017) successfully synthesized YBCO through the thermal treatment method, achieving impressive results with just a few readily available starting materials, such as metal nitrates, deionized water, and polyvinyl pyrrolidone (PVP) (Dihom et al., 2017; Dihom, Shaari, Baqiah & Chen, 2019). To our knowledge, no literature has been reported on the synthesising Bi-2212 using the thermal treatment method. As such, the current study aims to address this gap by producing Bi-2212 powders utilizing the thermal treatment method. This approach has the advantages of being simple and has the least by-product effluent compared to other methods (Dihom et al., 2017; Dihom et al., 2019; Kamari, Naseri, & Saion, 2014; Naseri, Saion, & Satayeshi, 2012).

Previous works demonstrated that introducing impurities into the Bi-2212 superconductor via doping, substitution, or addition improved both the superconducting and structural properties of Bi-2212 (Naseri et al., 2012; Masnita, Awang, & Abd-Shukor, 2022; Özcelik, Gürsul, Sotelo, & Madre, 2014; Suazlina, Yusainee, Azhan & Abd-Shukor, 2014; Zhang, Li, Hao, & Ma, 2015).This is attributed to the fact that impurities nanoparticles have smaller particle size that can settle more readily within the grains when added to the BSCCO system, resulting in enhanced properties (Zelati, Amirabadizadeh, Kompany, & Salamati, 2014). The incorporation of novel carbon-based nanomaterials, notably graphene (Abdullah, Kechik, Kamarudin, & Talib, 2023; Kamarudin et al., 2022), carbon nanotubes (CNT) (Fossheim, Tuset, Ebbesen, & Treacy, 1995; Galvan, Kim, Maple, & Hirata, 2000; Galvan, Li, Yuhasz, & Kim, 2004; Hannachi, Almessiere, Slimani, & Baykal, 2020), and carbon nanofibers (CNF) (Khalid, Kechik, Baharudin, & Kien, 2020), into superconducting materials such as YBCO, BPSCCO, Bi-2223, and Bi-2212 has garnered significant attention due to its potential to enhance their superconducting properties, particularly their critical temperature (T_c) and critical current density (J_c). Siregar, Yudanto, Chandra, & Lubis, (2021) have demonstrated the positive effect of incorporating 0.2 wt. % CNT into a sol-gel synthesized Bi-2223, resulting in an increase in T_c due to the introduction of defects (Siregar et al., 2021).

In this study, we aim to investigate the effect of carbon nanotube addition on the superconductivity of Bi-2212 superconducting properties and structural properties synthesised by the thermal treatment method.

Method

$Bi_2Sr_2CaCu_2O_9$ bulk superconductor was prepared by using a thermal treatment method with a stoichiometric of 2212; the metal nitrates of bismuth (III) nitrate, $Bi(NO_3)_3.5H_2O$ (Alfa Aesar 98%, UK), strontium nitrate, $Sr(NO_3)_2$ (Alfa Aesar 99.0%, UK), calcium nitrate tetrahydrate, $Ca(NO_3)_2.4H_2O$ (Sigma Aldrich 99.0%, USA), and copper (II) nitrate, $Cu(NO_3)_2.2.5H_2O$ (Thermo Scientific 98.0-102.0%, USA) were weighted accordingly to the stoichiometry. The powder precursors were dissolved in 300 ml of deionized water with 6g of polyvinyl pyrrolidone (PVP).

Prior to the dissolving of the precursors, bismuth nitrate was dissolved first in 30 ml of nitric acid. The solution was stirred for 2 hours at 80 °C on a hot plate. The solution was then dried in an oven overnight at 90 °C, producing a light green gel. The gel was crushed using a mortar and pestle into a fine powder. The powder underwent heat treatment at 600 °C for 4 hours followed by another round of treatment at 820 °C for 24 hours with intermediate grinding. Afterwards, the sample was further improved by the addition of carbon nanotube CNT (from Nanostructured & Amorphous Materials 95%, USA) and subsequently ground once more to guarantee thorough homogenisation. Then, mixed powders were pressed into circular pellets with a diameter of 13 mm and a 2 mm thickness and sintered at 840 °C for 48 hours. The constituent phases of the samples were characterised using X-ray diffraction (XRD) method using Phillips W 3040/60 Xpert Pro. The grain morphology of the samples was imaged using scanning electron microscopy, SEM (SEM-LEO 1455 VPSEM). Temperature-dependent resistance of the samples was measured using the standard four-point probe method with a closed-cycle helium cryostat.

Result and Discussion

The provided Figure 1 illustrates the X-ray diffraction (XRD) patterns of Bi-2212 samples containing varying weight percentages (wt.%) of carbon nanotubes (CNTs), ranging from 0.0 to 0.8 wt.%. This experiment serves to show the structural changes that occur in the Bi-2212 compound upon the inclusion of CNTs. The patterns showed a polycrystalline superconducting phase has been identified as a major phase, Bi-2212 (ICSD: 98-002-5079), with main peaks at hkl values of (1 5 1) and (0 0 2). All samples exhibited an orthorhombic crystal structure of Bi-2212, with a secondary phase recognised as Bi-2201. However, no CNT peaks were detected due to its minimal content. Table 1 shows the lattice parameters (*a*-, *b*-, and *c*-axis) and the volume fraction for phase formation of the samples. All the

results were obtained by refining the XRD data of the samples using the Highscore Plus software.

As outlined in Table 1, the inclusion of carbon nanotubes (CNT) resulted in an increase in the volume fraction of the Bi-2212 phase from 86.9% (0.0 wt.% of CNT) to 94.2% (0.2 wt.% of CNT). Further addition of CNT to 0.6 wt.% decreased the volume fraction of Bi-2212 to 84.8%, perhaps due to the increased appearance of impurities, specifically Bi-2201. However, the volume fraction of Bi-2212 phase increased back to 97.4% with the addition of 0.8 wt.% of CNT possibly due to enhanced particle orientation. The refined lattice parameters show that all the samples have an orthorhombic crystal structure. It can also be seen that the lattice parameters (*a*-, *b*-, and *c*-axis) remained relatively stable with increasing CNT percentages, suggesting that CNTs did not intercalate into the Bi-2212 grains.

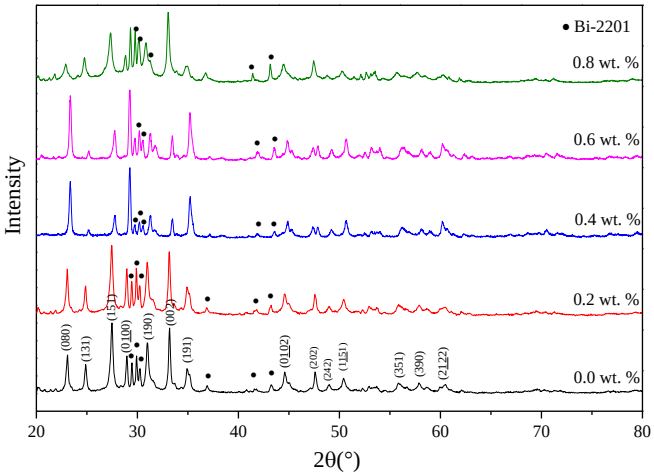

Figure 1. XRD pattern of Bi-2212 with CNT addition of 0.0, 0.2, 0.4, 0.6, and 0.8 wt.%

Table 1. Phase formation and the lattice parameters of the Bi-2212 superconductor

CNT addition (wt.%)	Orthorhombic unit cell			Volume fraction (%)	
	a (Å)	b (ÅÅ)	c (Å)	Bi-2212	Bi-2201
0.0	5.395	30.818	5.397	86.8	13.2
0.2	5.397	30.820	5.398	94.2	5.8
0.4	5.405	30.872	5.410	90.8	9.2
0.6	5.398	30.891	5.405	84.8	15.2
0.8	5.401	30.837	5.408	97.4	2.6

Figure 2 presents a normalized resistance versus temperature graph from 10 K to 250 K, illustrating the metallic behaviour exhibited by all samples between 80 K and 250 K. Except for the 0.8 wt.% CNT sample, which initially exhibited semiconductor behaviour. Notably, $T_{c\text{-onset}}$ values were influenced by CNT additions. From Table 2, it is evident that the value of $T_{c\text{-onset}}$ decreased from 75 K to 70 K for 0.0 wt.% and 0.2 wt.% samples, respectively. In contrast, the $T_{c\text{-onset}}$ value slightly increased to 76 K with the addition of 0.6 wt.% CNT before decreasing to 69 K with the addition of 0.8 wt.% of CNT. The inconsistency changed of $T_{c\text{-onset}}$ possibly due to sample inhomogeneity as evidenced by the relatively large breadth of transition, ΔT_c. The Bi-2212 sample with 0.6 wt.% CNT displayed the highest $T_{c\text{-onset}}$ and $T_{c\text{-offset}}$ at 79 K and 40 K, respectively, indicating a desirable enhancement in its superconducting properties. Meanwhile, sample with 0.2 wt.% CNT had the smallest ΔT_c, which means it had better crystallinity and grain connectivity, this observation is supported by the analysis of SEM images.

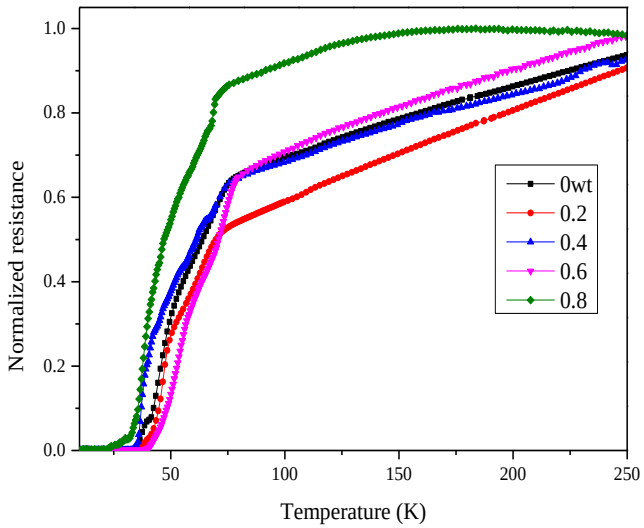

Figure 2. Normalized Resistance versus Temperature of Bi-2212 with CNT addition

Table 2. $T_{c\text{-onset}}$, $T_{c\text{-offset}}$, and ΔT_c for CNT addition of 0, 0.2, 0.4, 0.6 and 0.8 wt.%

Wt.%	0.0	0.2	0.4	0.6	0.8
$T_{c\text{-onset}}$ (K)	75	70	75	76	69
$T_{c\text{-offset}}$ (K)	36	36	35	40	31
ΔT_c (K)	39	34	40	39	38

Figure 3 shows the SEM images of Bi-2212 samples with varying weight percentages of CNT, taken at x5 K magnification, on the fractured surfaces of the pellets. All samples showed irregular shapes with randomly distributed grain orientation. The 0.4 wt.% CNT sample (Figure 3c) shows more voids between the grains compared to other samples, which affected the crystallinity of the samples. The average grain size of Bi-2212 is presented in Table 3 with the inclusion of CNTs. Increasing CNT content, decreased the average grain size of the samples, where the minimum value obtained was 1.018 µm for 0.4 wt.% sample, which contrasts with the previous study by Siswayanti, Yudanto, Imaduddin,

& Sebleku (2018). At a magnification of x50 K, Figure 4 confirms the dispersion of CNTs between the grains of Bi-2212 in the 0.2 wt.% CNT which aligns with the findings from Özçelik, Ergin, Depci, & Yavus (2019).

Table 3. The average grain size of the Bi-2212 with addition of carbon nanotubes.

Samples (wt. %)	Average Grain Sizes
0.0	1.247
0.2	1.030
0.4	1.018
0.6	1.020
0.8	1.050

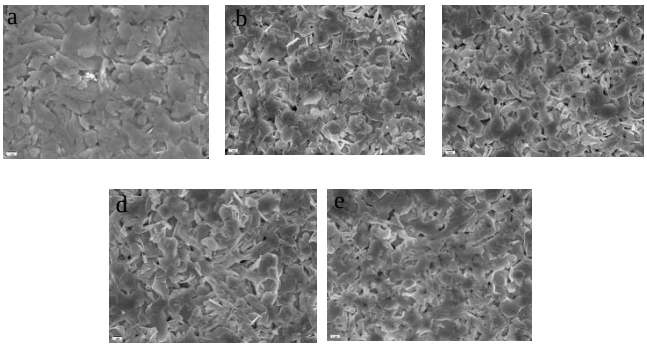

Figure 3. SEM surface morphology of CNT addition to Bi-2212 (a) 0.0, (b) 0.2, (c) 0.4, (d) 0.6, and (e) 0.8 wt. %

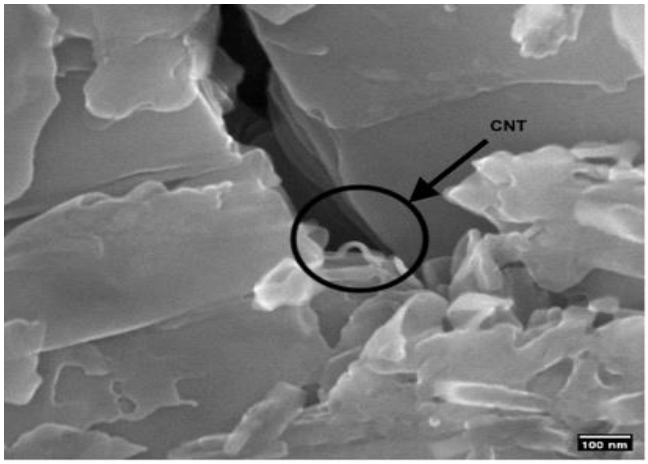

Figure 4. CNT spotted on the surface of BSCCO in 0.2 wt. % CNT addition under 50 K x magnification

Conclusion

This work focused on the study of phase formation and superconductivity of Bi-2212 bulks with addition of x wt.% CNT (x = 0.0, 0.2, 0.4, 0.6, 0.8 wt.%, respectively) prepared with thermal treatment method. XRD analysis showed that all samples had a main Bi-2212 phase with an orthorhombic structure, along with Bi-2201 phase impurities. The formation of Bi-2212 phase decreased with the addition of 0.2 to 0.6 wt.%, likely influenced by the addition of CNTs, which leads to the formation of impurities. However, no CNT peaks were detected due to their small amounts. Surface analysis revealed randomly distributed grains with occasional voids. In terms of superconducting properties, the CNT addition has an influence on increasing the T_c, potentially attributed to enhanced grain connectivity at this composition. The optimum value for the CNTs addition is 0.6 wt.% where T_c recorded the highest at 76 K.

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Author Contributions
Conceptualization: Safia Izzati Abd Sukor, Mohd Mustafa Awang Kechik; Methodology: Safia Izzati Abd Sukor; Formal analysis and investigation: Safia Izzati Abd Sukor, Mohd Mustafa Awang Kechik; Writing - original draft preparation: Safia Izzati Abd Sukor, Aliah Nursyahirah Kamarudin; Writing - review and editing: Mohd Mustafa Awang Kechik, Khairul Khaizi Mohd Shariff; Funding acquisition: Abdul Halim Shaari; Resources: Chen Soo Kien, Lim Kean Pah; Validation, Visualization and Supervision: Lim Kean Pah, Chen Soo Kien, Muhammad Kashfi Shabdin, Yazid Yaakob, Muhammad Khalis Abdul Karim.

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Conflicts of Interest
The authors declare no conflict of interest.

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