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# INFLUENCE OF HEAT TREATMENTS ON ELECTRICAL PROPERTIES AND MICROSTRUCTURE OF 10% MASS FRACTION OF SUCROSE YBCO SUPERCONDUCTOR

## (Kesan Rawatan Haba Ke Atas Sifat-Sifat Elektrik dan Mikrostruktur 10% Pecahan Jisim Sukrosa YBCO Superkonduktur)

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**Abstract**

The influence of different heat treatments on the superconducting properties of 10% mass fraction of sucrose structure YBCO superconductor was investigated. X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) equipments were used to determine the phase of superconductor and structural studies respectively at10% mass fraction of sucrose. The samples were prepared via solid state (SSM) and co-precipitation (CPM) reaction methods and underwent sintering and heat treatment process at 900°C, 930°C and 960°C respectively with mixing of $C_{12}H_{22}O_{11}$ sucrose during pelletization. The $T_{C,on}$ decreases with respect to higher heat treatment temperature. The suppression of both $T_{C,on}$ and $T_{C,off}$ indicates the destruction of superconductivity trends. The best $T_{C,off}$ were achieved in pure SSM and CPM samples sintered at 950°C for 5 hours with $T_{C,off}$ 86K and 91K respectively. Comparing with pure YBCO, the 10% mass fraction of sucrose YBCO exhibited higher critical current, $I_C$ by two times. It indicates the effect of high surface area in porous structure. The XRD results confirmed that all the samples remain in single phase, which indicates no effect of sucrose in the porous structures sample and maintaining in orthorhombic structure. Higher heat treatment at 960°C resulted in destruction on its superconductivity behavior due to the partial melt phase on its microstructure, especially in CPM. This is due to the smaller grain size of samples which trapped more heat and causing partial melting to occur rapidly. It can be deduced that, annealing temperatures at 900°C and 930°C are the best optimum heat treatments for CPM and SSM porous superconductor, respectively.

**Keywords**: YBCO superconductor, porous superconductor, sucrose

**Abstrak**

Kesan pelbagai rawatan haba ke atas sifat-sifat 10% pecahan jisim sukrosa ke atas YBCO superkonduktor telah dikaji. Peralatan seperti "X-Ray Diffraction" (XRD) dan "Scanning Electron Microscopy" (SEM) telah digunakan untuk menentukan fasa superkonduktor dan strukturnya pada 10% pecahan jisim sukrosa. Bahan ujikaji telah disediakan melalui kaedah keadaan pepejal (SSM) dan pemendakan sampingan (CPM), melalui pembakaran dan proses rawatan haba pada suhu 900°C, 930°C dan 960°C dengan mencampurkan sukrosa $C_{12}H_{22}O_{11}$ semasa proses pembentukkan pelet. $T_{C,on}$ , suhu genting onset menurun dengan kenaikan suhu rawatan haba. Penurunan kedua-dua $T_{C,on}$ dan $T_{C,off}$ menunjukkan penghapusan arah aliran kesuperkonduktoran. $T_{C,off}$ yang terbaik dicapai pada sampel tulen SSM dan CPM yang dibakar pada 950°C selama 5 jam di mana $T_{C,off}$ adalah masing-masing 86K dan 91K.Apabila dibandingkan dengan YBCO tulen, 10% pecahan jisim sukrosa YBCO mempunyai arus genting, $I_C$ yang dua kali lebih tinggi. Ia menunjukkan kesan luas permukaan yang tinggi dalam struktur berpori. Keputusan XRD mengesahkan bahawa semua bahan ujikaji kekal dalam satu fasa,yang menunjukkan sukrosa tidak membawa kesan ke atas sampel dan sampel kekal dalam struktur ortorombik. Rawatan haba yang tinggi pada 960°C mempamirkan kerosakan pada sifat kesuperkondutorannya akibat fasa separa cair pada mikro sturktur terutamanya pada CPM. Ini disebabkan oleh pengecilan saiz butir sampel yang memerangkap lebih banyak haba dan menyebabkan proses separan cair berlaku pada kadar yang tinggi. Ini boleh disimpulkan bahawa suhu pembakaran pada 900°C dan 930°C masing-masing adalah suhu optimum rawatan haba untuk CPM dan SSM pori superkonduktor.

**Kata kunci**: YBCO superkonduktor, pori superkonduktur, sukrosa

## Introduction

Although its ability as a high field magnet has been realized, still, bulk YBCO loses its high temperature superconducting property due to low critical current densities. Despite critical current density ($J_C$) of thin films superconductor, for instance, exceeds bulks by at least two orders of magnitude, thin films also are not really suitable for high integral currents [1].Therefore, the applications of superconductor require material to be available in a variety of shapes and forms. Bulk and thin films superconductor reveal their own strength and weaknesses. The discovery of intermediate forms such as foams and porous superconductor has attracted many researchers to study its unique properties that are highly interesting for our modern applications. The porous pattern is believed to be the best solution for the low critical density's problems [1-6].Foams and porous superconductor have common properties where contained open pore structures that is believed to improve the performance of the superconductor which is not in bulks and thin films. Many other aspects are related to foam and porous structure, for example, more efficient heat transfer, faster oxygenation, less related micro cracking, possibility of reinforcement and of interlocking connections [2]. Although many interesting properties can be found in open porous structure, but one of the most unique properties is that it can results in higher $J_C$. Even though there are numbers of publications concerning methods of creating open porous superconducting structures [3,4], none focuses on its critical current. High critical current is important for various applications. Thus, this research will focus on investigating YBCO porous superconductor's unique properties and to investigate whether heat treatment and level of porosity will influence the YBCO superconducting properties, mainly on its critical current ($I_C$). Structural studies of the porous superconductor using powder X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) reveal the relationship between superconducting properties and the YBCO's porous structure and microstructure.

## Materials and Methods

For YBCO solid state method (SSM) sample, the precursor was prepared using powders of yttrium oxide, $Y_2O_3$ (Alfa Aesar, 99.90%), barium carbonate, $BaCO_3$ (Alfa Aesar, 99.80%) and copper oxide, CuO (Aldrich, 99.00%), and carefully mixed thoroughly according to its stoichiometry, $YBa_2Cu_3O_{7-x}$ in a ratio of 1: 2: 3. Then, the chemicals in grey powders were ball-milled for 24 hours to obtain fine powders and after dried in an oven for several hours. Then, the dried mixture was ground in a mortar and pestle. The mixture was then sintered at 900°C and 950°C for 5 hours respectively to get powder sample of SSM.

Co-precipitation method (CPM) sample was prepared from an aqueous solution of the salts of metals using an oxalate precipitation reagent under basic pH conditions. In this technique, a simultaneous coprecipitation of Y, Ba, and Cu oxalates by precipitating in mixture of ethanol: water (70:30) medium and using triethylammonium-oxalates (TEO) as the precipitating agent under controlled pH conditions. Stock solution of barium nitrate, Ba $(NO_3)_2$ (Aldrich, 100%), copper nitrate, $Cu(NO_3)_2$ (Hamburg chemicals, 99.90%) and yttrium nitrate, $Y(NO_3)_3$ (Aldrich, 99.90%) were prepared by dissolving the corresponding metal nitrates in distilled water. The precipitating agent, TEO $((C_2H_5)_3NH)_2C_2O_4$ was prepared by dissolving triethylamine (TEA) (Sigma-Aldrich, 99.00%) and oxalic acid (Merck, 99.00%) (2:1 mol ratio) in ethanol (System, 99.00%). After mixing the metal nitrate solution (1:2:3) in a beaker, the pH of the solution was adjusted to 4.0 by 5% solution of TEA in 70:30 ethanol:water mixtures resulting in a bluish color precipitate appeared. The solution was stirred for 30 minutes and then filtered. The crystalline precipitate was washed with ethanol and dried in an oven at 300°. The mixture was then sintered at 900°C and 950°C for 5 hours respectively to get powder sample of CPM.

Then, pressed the powder samples of SSM and CPM at 30MPa to form a pure sample pellet. To create an open pores superconductor, the crystalline sucrose (HmBg Chemicals, 99.00%) as filler was added in an amount necessary to obtain 10% mass fraction of sucrose using the mass fraction formula as in equation (1). The pores samples were then pressed to 30MPa to form pellet.

In the mass fraction formula,

$$W_A = \frac{M_A}{M_{tot}} \times 100\% \quad (1)$$

where,

$W_A$ = mass fraction of one substance
$M_A$ = mass of sucrose, (g)
$M_{tot}$ = total mixture of mass (sucrose + YBCO powder), (g)

Then, the 10% mass fraction of sucrose samples of both SSM and CPM were heated at 400°C in order to burn out the sucrose completely before undergoing three different heat treatments which are 900°C, 930°C and 960°C at 5 hours respectively. The pure samples are also subjected to the same annealing heat treatment conditions for comparison.

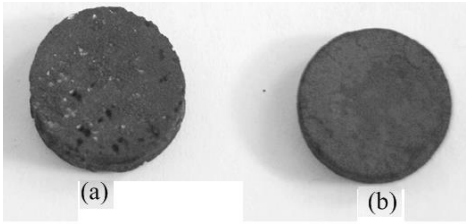



Figure 1. Different porosity of (a) 10% mass fraction of sucrose and (b) non-porous samples after underwent heat treatment.

Sample characterization process involves the electrical and structural aspects of these samples as well as its density measurement. Measurement of electrical resistance was made using four-point probe method whereas the structural and microstructural characterizations were made using X-Ray Diffraction (XRD) and Scanning Electron Micrographs (SEM).

X-Ray Diffraction (XRD) is used to study the crystal structure, particle size, phase formation and to a lesser extent, crystal defect and disorder. While in this research, XRD is used extensively in order to study the phase formation of porous YBCO in powder and pellet forms with absence of remaining sucrose filler after heated. The lattice parameter *a*, *b*, and *c* were determined using this technique. In a typical XRD, an X-Ray tube provides copper-Kα radiation that is collimated through an aperture diaphragm before the Kα radiation is absorbed by a 12μm thick nickel filter and it is then detected by standard scintillation counter. The sample rotates by a consecutive step process such that an angel of incidence of the primary beam gradually increased between present values. The detector is correspondingly moved around the sample at precise double the angular velocity ensuring that at all time the diffraction angle (2 θ) is twice the glancing angle (θ). The control of sample position and scintillation data are controlled and recorded by a computer where a real time display of the data can be viewed. Data collection times were taken in 2 or 3 hours and the scanning range 2θ is between 2° and 60°.

The porosity structures of samples are characterized by the calculation of density samples using Archimedes Principle as in equation (2) to ensure the porosity pattern.

$$\rho_s = \frac{m_s \times \rho_w}{m_w} \quad (2)$$

where,

$\rho_s$ = density of sample
$\rho_w$ = density of water, ($g/cm^3$)
$m_s$ = mass of sample
$m_w$ = mass of water

The dc resistivity measurements of the samples are performed on the sample between 20K and 300K with the four-probe method to obtain its critical temperature ($T_C$). The critical current ($I_C$) was also measured using the four-point probe technique. The samples was placed in a sample holder of a Cryogenic Closed Cycle cryostat by using Electrolube silver paint to allow the current flow and minimize the contact resistance, and mounted with four probes using copper wires to connect the samples to the device. The pellets were lightly polished using sandpaper before mounting to remove foreign particles and dust. A d.c current was supplied by Keithley Model 197A Autoranging Multimeter with an accuracy of $\pm$0.001 mV. The temperature of cryostat is controlled by Lakeshore Model 340 temperature controller ($\pm$0.001K). For each sample, three different current were applied and the corresponding voltages were recorded for $I_C$ measurement. The average value of resistivity was then taken as the resistivity at 300K (room temperature).

In addition, SEM is used to study the surface of samples at very high magnification and to obtain the micrographs to give clues how the surface morphology of the samples were like. To operate SEM, samples were placed on a carbon-conductor tape on the holder. The sample is mounted inside the main chamber where the holder is attached to the main platform by sliding it to fit nicely to the holder mount. Fine electron beam is aimed at the surface of sample. The intensity of the reflected beam is then picked up by the sensor. This signal is then fed to the computer which maps the intensity of the electron reflection and thus would produce a black and white picture. For the purpose of this study, the images of the samples were magnified 15,000 times at accelerating voltage of 5 kV.

Grain size may be determined by using quadrant technique. In this method, the straight line are drawn and measured through the grains. The measured length are then is compared with the grain scale given in the micrograph pictures which is 1µm actual size of grain = 0.8 cm size of grain in the micrograph picture.

## Results and Discussion

The result and discussion on this research contains results from resistance measurement, X-Ray diffraction analysis (XRD) and scanning electron microscope (SEM) micrographs from all samples, from pure to 10% mass fraction of sucrose sample, where sucrose is added after sintering at 900°C and 950°C, and then heat treated at 900°C, 930°C and 960°C at 5 hours respectively.

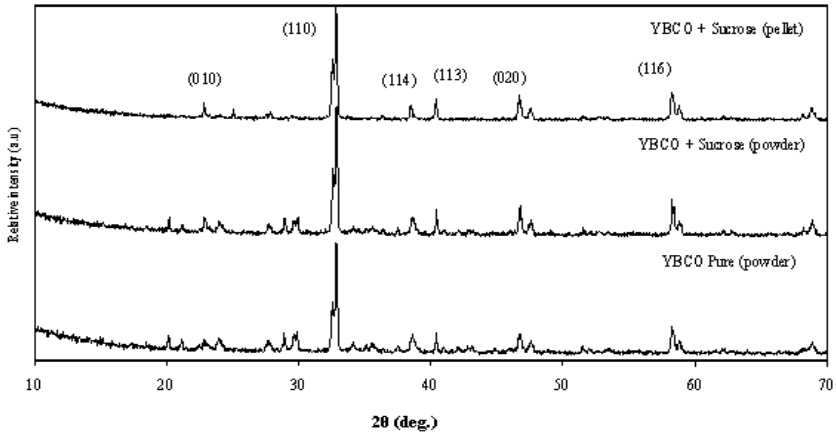



Figure 2. XRD pattern for pure YBCO and 10% mass fraction of sucrose YBCO after heated at 400°C and annealed at 930°C via CPM.

Table 1. Lattice parameter of pure and porous YBCO samples via CPM.

| Sample YBCO | a (A°) ± 0.1% | b (A°) ± 0.1% | c (A°) ± 0.1% | Volume ($A°m^3$) (± 0.3%) |
|---|---|---|---|---|
| YBCO + Sucrose (pellet) | 3.824 | 3.889 | 1.687 | 1.738 |
| YBCO+Sucrose (powder) | 3.818 | 3.890 | 1.668 | 1.732 |
| YBCO (pure) | 3.820 | 3.887 | 1.651 | 1.730 |

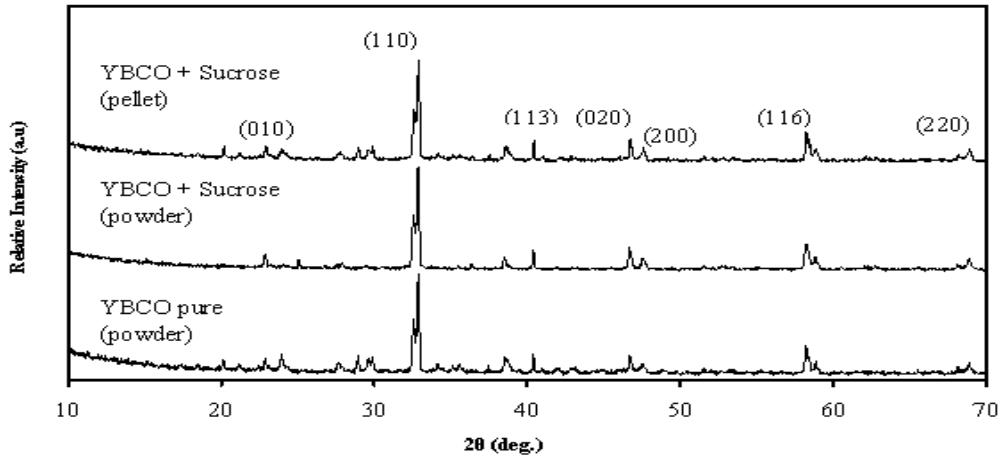



Figure 3. XRD pattern for pure YBCO and 10% mass fraction of sucrose YBCO after heated at 400°C and annealed at 930°C via SSM

Table 2. Lattice parameter of pure and porous YBCO samples via CPM.

| Sample YBCO | a (A°) ± 0.1% | b (A°) ± 0.1% | c (A°) ± 0.1% | Volume ($A°m^3$) (± 0.3%) |
|---|---|---|---|---|
| YBCO + Sucrose (pellet) | 3.827 | 3.893 | 1.699 | 1.743 |
| YBCO+Sucrose (powder) | 3.888 | 3.825 | 1.662 | 1.734 |
| YBCO (pure) | 3.820 | 3.887 | 1.651 | 1.730 |

Figure 2 and 3 above presents the XRD patterns for a reference sample (pure sample) and porous sample, after adding sucrose, heated at 400°C and annealed at 930°C for 5 hours. All samples showed a degree of single phase 123 where the $T_{c,on}$ are about 73K and 88K.

The X-Ray analysis also succeeds revealing that this orthorhombic structure of superconductor shows the absence of the remaining sucrose filler after heated. It is obviously shows that the sucrose in the YBCO samples was burned out completely.

Adding sucrose as filler had induced an increasing in surface area on open porous YBCO structures and hence reduces its density values. As shown in Table 1and Table 2, the inter-plane *c* changed practically as compared with *a* and *b* lattice. It is known that porosity had reduced the $T_C$ and increased the unit cell volume. It is believed that the loss of oxygen content resulted in the expansion of *c* lattice. For these porous samples, it can be observed that with decreasing $T_C$, *a*-lattice parameter was not much affected but *c*-lattice values are increased.

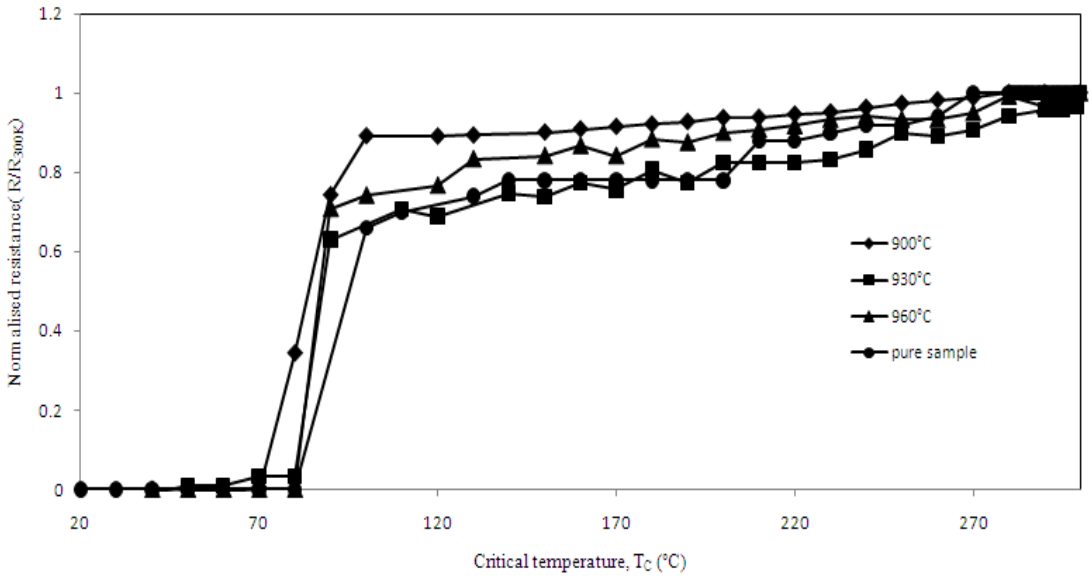



Figure 4. $T_C$ for pure and 10% mass fraction of sucrose YBCO superconductor after annealing at 900°C, 930°C and 960°C via CPM.

The measurement of electric resistance characteristic for pure and porous samples showed metallic behavior of electric conductivity above the onset critical temperature ($T_{C,on}$ )for all samples as seen in Figure 3 and 4. This shows that adding sucrose as filler does not alter the superconducting behavior of the pure YBCO superconductor but alter the electrical properties of superconductor. The highest value of onset critical temperature ($T_{C,on}$) and offset critical temperature ($T_{C,off}$) were achieved on a sucrose-free sample. For 10% mass fraction of sucrose YBCO superconductors via CPM with 900°C heat treatment is found to have the highest $T_C$ where $T_{C,off}$ = 77K and $T_{C,on}$ = 86K. Generally, the same trend for all heat treatment series for both methods showed that the critical transition temperature ($T_C$) value as illustrated in Table 2 and 3 in porous superconductor is considerably decrease with increased in porosity.

Table 3. $T_{C,off}$, and $T_{C,on}$, and for pure and 10% mass fraction of sucrose YBCO heat treated sample at 900°C , 930°C and 960°C via CPM and SSM.

| Heat treatment / Method | Porosity % | $T_{C,off}$ K ± 1 | $T_{C,on}$ K ± 1 | ΔT K ± 1 |
|---|---|---|---|---|
| Pure CPM | 0 | 91 | 97 | 6 |
| 900°C / CPM | 10 | 77 | 86 | 9 |
| 930°C / CPM | 10 | 67 | 85 | 18 |
| 960°C / CPM | 10 | 70 | 100 | 30 |
| Pure SSM | 0 | 86 | 90 | 4 |
| 900°C / SSM | 10 | 61 | 88 | 27 |
| 930°C / SSM | 10 | 73 | 88 | 15 |
| 960°C / SSM | 10 | 54 | 71 | 17 |

The effect of three different heat treatments suggested that the general performances of porous superconductor failed to improve the values of its critical temperature. Probably, the oxygen inside YBCO lattice suppressed the superconducting properties as suggested by previous researchers [7]. However, in pure YBCO samples, the remaining of copper oxide (CuO) along the [0 1 0] direction in the samples instead of YBCO itself did not cause any significant decrease in $T_C$. Nevertheless, in porous samples, an apparent reduction of $T_C$ is observed and some of the sample has lower than expected $T_C$ such as in the 10% porous YBCO. It could be due to the porous samples having an open porous structure resulting in loss vital oxygen from its structure.

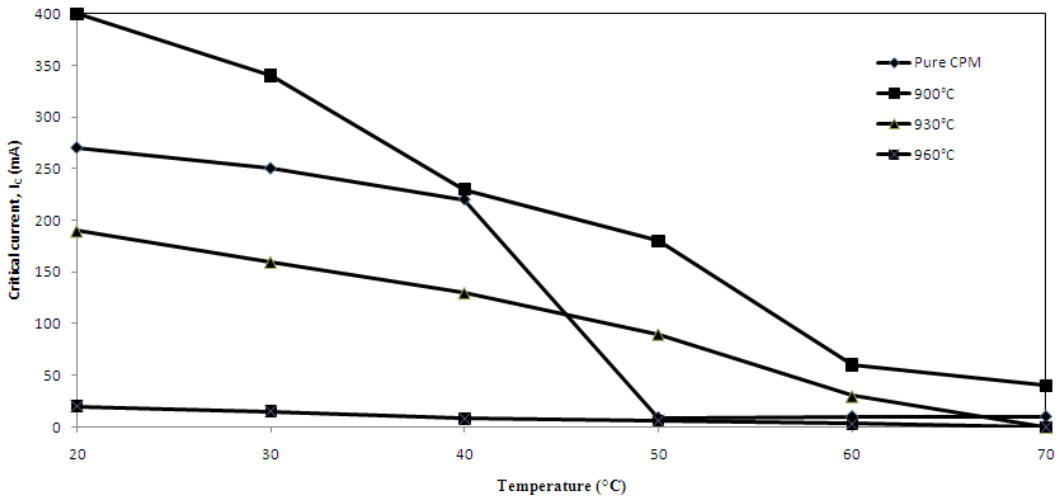



Figure 5. Critical current, $I_C$ for 10% mass fraction of sucrose YBCO at 20K to 70K for samples heat treated at 900°C, 930°C and 960°C via CPM.

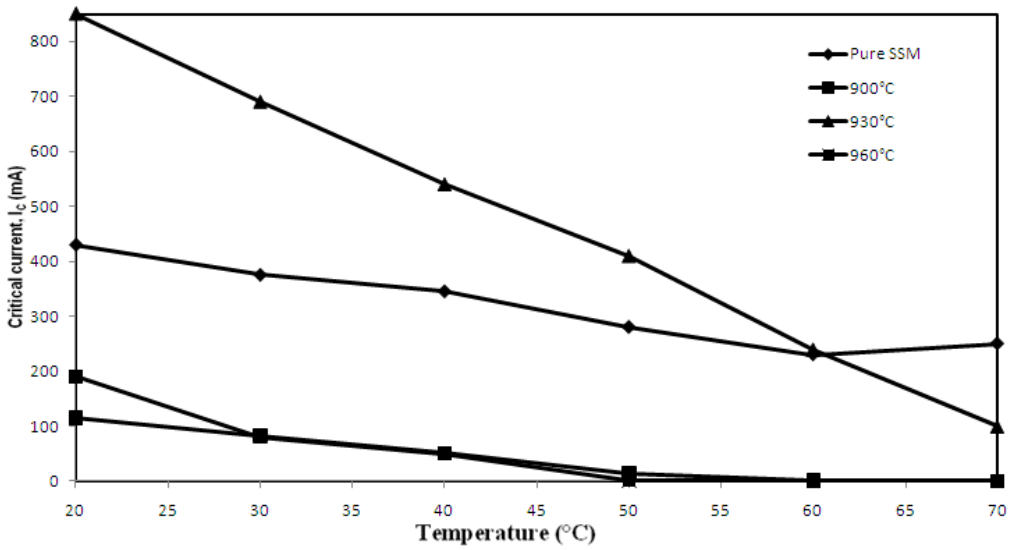


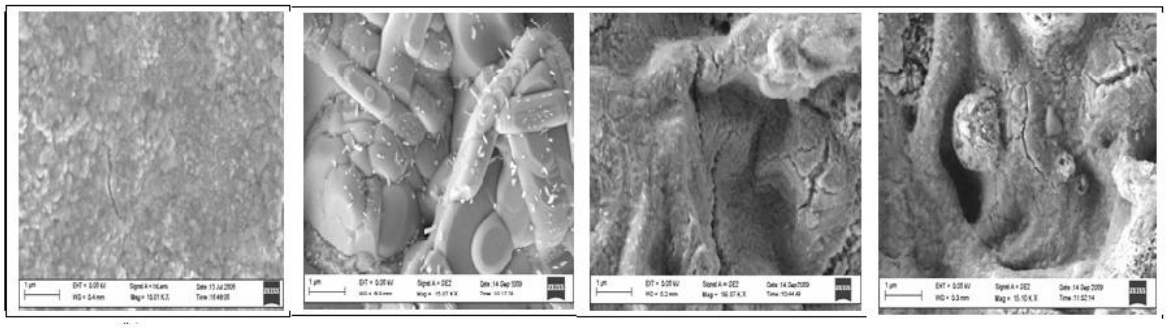

Figure 6. Critical current, $I_C$ for 10% mass fraction of sucrose YBCO at 20K to 70K for samples heat treated at 900°C 930°C and 960°C via SSM.

Table 4. Critical current, $I_{C,}$ for pure and 10% mass fraction of sucrose YBCO heat treated sample at 900°C , 930°C and 960°C via CPM and SSM.

| Heat treatment / Method | Critical current at various temperature (mA) | | | | | |
|---|---|---|---|---|---|---|
| | 20K | 30K | 40K | 50K | 60K | 70K |
| Pure CPM | 270 | 250 | 200 | 90 | 10 | 10 |
| 900°C /CPM | 400 | 340 | 230 | 180 | 60 | 40 |
| 930°C /CPM | 190 | 160 | 130 | 90 | 30 | - |
| 960°C /CPM | 20 | 15 | 8 | 6 | 3 | - |
| Pure SSM | 430 | 375 | 345 | 280 | 230 | 250 |
| 900°C / SSM | 115 | 83 | 51 | 14 | 1 | - |
| 930°C / SSM | 850 | 690 | 540 | 410 | 240 | 100 |
| 960°C / SSM | 190 | 80 | 50 | - | - | - |

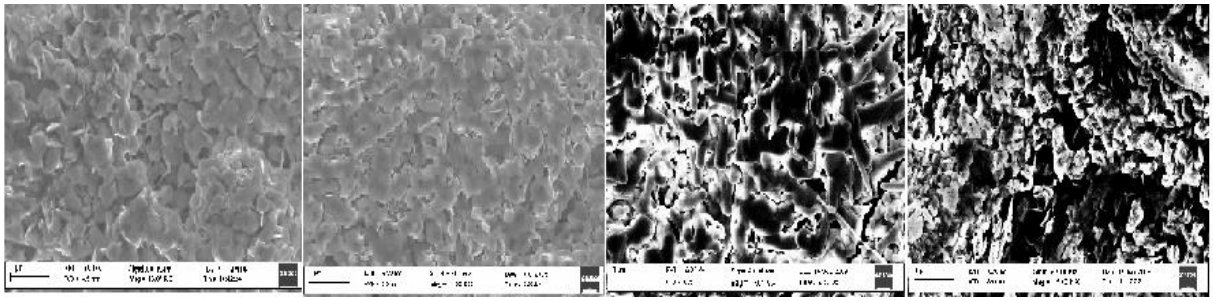
Critical current for superconductivity samples also depends on the temperature. Lower temperature of heat treatment's sample will result in higher critical current in the sample as seen in Table 4. Comparing with pure samples, the 10% mass fraction of sucrose YBCO is able to transport significantly higher critical current than the dense structure due to the increase in the surface area of porous YBCO as illustrated in Figure 5 and 6.

The $I_C$ of samples increases in increasing porosity from pure to 10% mass fraction of sucrose for certain heat treatments. The increase of $I_C$ may be due to the thin grain boundary and growth of grains on the sample structures as observed in 10% mass fraction of sucrose YBCO for 900°C heat treatment (CPM) and 930°C heat treatment (SSM). This porosity resulted in larger grain size hence, higher in $I_C$ compared to others as seen in Figure 7 and 8. The existence of large, elongated grains separated by low-angle grain boundaries can increase the $I_C$ value since it is capable to transport large amounts of currents [8]. Higher heat treatment at 960°C resulted in partial melting in the samples and thus degraded the $I_C$ and affects its superconductivity. With increasing sintering temperature, the total number of grains will increase and consequently, the total number of intergrain weak links increases [9]. It can be obviously seen in Figure 7 to 8 that 960°C heat treatment led to microcracks presence in the sample and causes considerable reduction in $I_C$ value [10].



(a) (b) (c) (d)

Figure 7. Micrographs of fractured surface of (a) pure and 10% mass fraction of sucrose YBCO sample that underwent heat treatment for (b) 900°C, (c) 930°C and (d) 960°C respectively via CPM.



(a) (b) (c) (d)

Figure 8. Micographs of fractured surface of (a) pure and 10% mass fraction of sucrose YBCO sample that underwent heat treatment for (b) 900°C, (c) 930°C and (d) 960°C respectively via SSM.

Different heat treatments altered the grain size and texturing. However, these samples showed a very compact structure with smaller grain size approximately in the order of 0.25 µm with addition of sucrose the grain size increase to ~ 2 µm. Pure sample tends to have higher $T_C$ and smooth curves (Figure 4, 7 and 8).

Different heat treatments altered the grain size and texturing. However, CPM samples showed a very compact structure with smaller grain size approximately in the order of 0.25 µm with addition of sucrose the grain size increase to ~ 2 µm. Thus, CPM samples have finer size compared to the SSM samples. Pure CPM sample tends to have higher $T_C$ and smooth curves when compared to SSM sample as seen in Figure 4.

For the 900°C heat treatment samples, the effect of sucrose addition caused the formation of porous structure. It can be concluded that, higher temperature of heat treatment up until 960°C produced smaller grain size samples and caused the partial melting occurred hence destroyed the structure of the samples. Heat treatment of 930°C is the optimum heat treatment for porous samples where it's $T_{C,on}$ and $T_{C,off}$ are almost similar to pure sample. The 10% mass fraction of sucrose enhanced grain growth and orientation in all the samples.

## Conclusion

The effect of heat treatment on percentages of porous YBCO superconductor via SSM and CPM has been investigated. It can be deduced that heat treatment and porosity play important role in superconductivity of YBCO. The best superconducting behavior in terms of the critical temperature was observed in pure YBCO with no addition of sucrose as filler especially in CPM samples. The CPM method meanwhile offers some advantages over SSM such as simple to prepare and allows a rapid formation of the YBCO phase. Moreover, in CPM the particle size is easy to control and the stoichiometry ratio can be determined and thus, it showed that the CPM method produced a very fine powder. It is proved that the critical temperature decreases when the density of superconductor decreases which is in increasing of porosity. No improvement in the critical temperature behavior was observed in porous structure superconductor. However, an obvious difference between porous and non-porous superconductor is the effect of surface that is much greater in porous superconductor and hence resulted in increase of critical current up to twice as much higher compared to non-porous superconductor in for 900°C heat treatment (CPM) and 930°C heat treatment (SSM). XRD pattern showed that having sucrose as a filler phase have no effect on its structure for both CPM and SSM. It is proved that the crystallographic structure remain in orthorhombic where a, b, and c lattice were not much affected. However, higher heat treatment, 960°C will results in destruction on its superconductivity behavior due to the partial melt phase to the smaller grain size of CPM samples easily trapped more heat and caused partial melting to occur. It also led to microcracks presence in the sample and causes considerable reduction in $I_C$ value. For this reason, it can be concluded that, heat treatment of 900°C and 930°C are the optimum heat treatment for CPM and SSM porous superconductor respectively.

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