# Investigation of Electrical Properties and Crystal Structure of $Y_5Ba_8Cu_{13}O_x$ Prepared by Melt Process

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## Abstract

Samples of Y5-8-13 superconductors were synthesized by the melt process. Average critical temperature was about 91 K. The sample had two compounds: superconducting compounds and non-superconducting compounds. The percentages of superconducting compounds and non-superconducting compounds were 30 % and 70%. The lattice parameter of the first compound was *a*=3.82912 Å, *b*=3.88788 Å and *c*=48.46370 Å. The second compound separated in two types, with percentages of 35% and 35% of Y211 ($Y_2BaCuO_5$) and $BaCuO_2$, respectively. The Y211 had lattice parameters of *a*=7.21450 Å, *b*=12.23320 Å and *c*=5.62140 Å, and $BaCuO_2$ was lattice parameter *a*=*b*=*c*=18.23560 Å. The melt samples reduced the non-superconducting compound, and the surface was homogenous.

**Keywords:** Melt process, Y-based superconductors, critical temperature

## 1. Introduction

In 2010, Udomsamuthirun *et al*. [1] synthesized a new YBaCuO superconducting material by conventional solid state method. The assumption of synthesis was Y-atoms plus Ba-atoms equal Cu-atoms. The new YBaCuO consisted of Y3-8-11, Y5-8-13, Y7-11-18 and Y13-20-33. In 2011, Sujinnapram *et al*. [2], investigated crystal structure of this material by powder X-ray diffraction and characterized the phase composite by Reitveld refinement method [3]. The results revealed that the samples consisted of superconducting compounds and non-superconducting compounds. The Y5-8-13 had the most non-superconducting compound. However, critical temperature and XRD pattern of the Y5-8-13 were close to Y123. The *c* lattice parameter of Y5-8-13 superconductors was 5x longer than the *c* lattice parameter of Y123. So, to improve the physical properties of this materials, good preparation to reduce the non-superconducting compound was required. The melt textured [4] was an alternative process widely used in preparation of superconductors to improve the physical properties of this material.

In 2015, Bortolozo *et al*. [5], synthesized a Y123 by melt texture and doping Ta of 2 wt% and 4 wt%. The precursors of the material were $BaCO_3$, $Y_2O_3$ and CuO. The dimension of the samples was 10x10 $mm^2$ and 2 mm in thickness. The samples were calcined at 900 $^oC$ in air for 24 hrs and repeated twice. The sintering temperature was 1,200$^oC$ for 48 hrs. The critical temperature of samples was measured by the four-probe method with a temperature range between 70 K-200 K. The critical temperature was about 90 K. In 2008, Jongprateep and Dogan [6] synthesized

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Y123 by the melt process method with doped Nd of 0.1% to 0.5%. Samples were calcined at 990ºC and sintered at 1,050ºC for 0.5 hrs. The crystal structure was investigated by powder X-ray diffraction. The lattice parameter of the *c* axis increased from 11.6796 A to 11.7077 Å with increased Nd concentration.

In 2000, Yamashita *et al*. [7] synthesized a bulk sample and thin film of Y123 by the co-precipitation method and added the second phase of Y123 was $Y_2BaCuO_5$ (Y211). The temperature of sintering was at 915 ºC and partially melted at 1100ºC for 0.3 hrs. The critical current density was measured at 77 K, The sample of sintering and the partially melt showed the current density at 1,000 A/cm$^2$ and 3,000 A/cm$^2$, respectively. It can be seen that the melt process improved the basic physical properties of these materials.

In this paper, we synthesized Y5-8-13 by the melt textured method. Physical properties of our samples were investigated by a conventional standard four-probes method. The crystal structure was investigated by powder X-ray diffraction. The elemental compositions were carried out by EDX (Energy Dispersive X-ray spectroscopy).

## 2. Materials and Methods

Pellets of Y5-8-13 were synthesized from high-purity of $Y_2O_3$, $BaCO_3$ and CuO. The powder was mixed in an alumina crucibles for 30 mins. The mixed powder was calcinated at 950 ºC for 24 hrs as shown in figure 1. Black solidification of the mixed powder was obtained. The powder was reground into 150 μm.

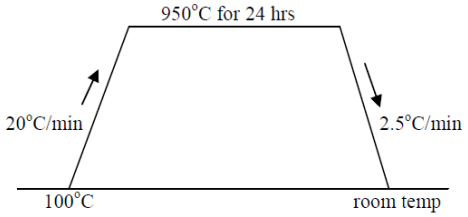


**Figure 1.** Schematic of the calcinations

The fine powder was pressed under pressure of 2,000 psi into pellets 30 mm in diameter and 3 mm in thickness. For the first sintering process, the starting temperature was 100 ºC. The temperature then was increased with a rate of 20ºC/min until reaching 950 ºC. The pellets then were kept constant at 950 ºC for 12 hrs. Temperature was increased again at the rate of 2.5 ºC/min until reaching 1,030 ºC. At 1,030 ºC the temperature was kept constant for 30 mins. The samples then were cooled down at the rate of 2.5 ºC/min until the temperature went down to 950 ºC. The samples were kept at 950 ºC for 12 hrs then cooled down again at the rate of 2.5ºC until the temperature went down to 500 ºC. The samples were kept at 500 ºC for 12 hrs then cooled down again at the rate of 2.5 ºC/min until reaching room temperature as shown in figure 2.

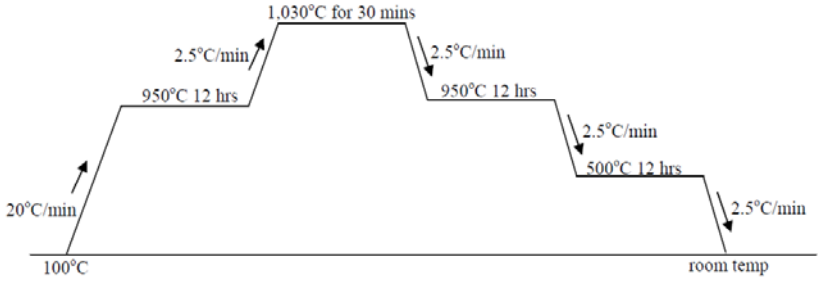


**Figure 2.** Schematic of the sintering

The obtained bulk samples were characterized for physical properties. The critical temperature measurement was carried out by the four-probe method with the d.c. constant current at 200 mA, 250 mA and 300 mA, respectively. The measurement temperature ranged between 77 K-120 K to observe the exchange superconducting state and normal state. The composition and lattice parameters were investigated by powder X-ray diffraction (Scintag XDS 2000 Diffractometer, Phillips X-Pert), over angle ranging in 2θ from 10° to 90° and Reitveld method characterized the crystal structure. The elemental compositions, microstructure and elementary spectra analysis of the samples were revealed by EDX (Energy Dispersive X-ray spectroscopy, FEI Quanta 400) using mapping techniques.

## 3. Results and Discussion

The curve between electrical resistance versus temperature is shown in figure 3. The values of the $T_c$ offset (K), $T_c$ middle (K), $T_c$ onset (K) and average $T_c$ (K) are shown in Table 1. The curve of the measurement showed two steps. The first step was superconductivity state occurrence at 77 K until the offset temperature ($T_c$ offset). The $T_c$ offset was the endpoint of the superconductivity and defined the resistance of the samples at zero. The temperature increased until $T_c$ middle (K). The $T_c$ middle (K) was 50% interval $T_c$ offset (K) and $T_c$ onset (K). Until the temperature reached the $T_c$ onset point, the sample showed a normal state which the temperature increased to 120 K. The d.c. constant current of 200 mA, 250 mA and 300 mA were used simultaneously for the measurement as shown in the blue solid line, red solid line and black solid line, respectively. The highest $T_c$ offset, $T_c$ middle and $T_c$ onset were recorded at the current of 200 mA.

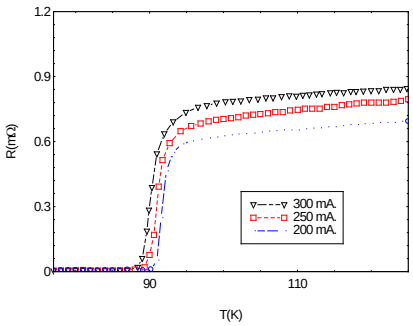


**Figure 3.** Electrical resistance dependence temperature of the melt Y5-8-13 superconductors

**Table 1.** The constant current with various of critical temperature.

| Current (mA) | $T_c$offset (K) | $T_c$middle (K) | $T_c$onset (K) | Average $T_c$ (K) |
|---|---|---|---|---|
| 200 | 89.06 | 91.10 | 93.14 | 91.10 |
| 250 | 87.99 | 90.34 | 92.70 | 90.34 |
| 300 | 86.99 | 89.49 | 91.98 | 89.44 |

The fine powder was characterized for the lattice parameter of the phase composition by powder X-ray diffraction. The Reitveld refinement was used to determine percentage and lattice parameters of phase compositions. The result of the XRD pattern of Reitveld analysis is shown in Figure 4. The samples can be separated into two compounds, The first compound was the percentage of 30% for superconducting phase with the lattice parameters as *a*=3.82912 Å, *b*=3.88788 Å and *c*=48.46370 Å. The second compound was non-superconducting phase was 70% and The non-superconducting compound can be separated into 2 types, The first type of 35% for Y211 ($Y_2BaCuO_5$) had the lattice parameters as *a*=7.21450 Å, *b*=12.23320 Å and *c*=5.62140 Å and the second type of 35 % for $BaCuO_2$ had the lattice parameters as *a*=*b*=*c*=18.23560 Å. From the results of Sujinnapram *et al*. [2], the Y5-8-13 synthesized by solid state reaction showed a percentage superconducting compound of 28 % with the lattice parameter as *a*=3.81909Å, *b*=3.89788 Å and *c*=50.46070Å. The non-superconducting compound had 3 types which were Y211 12%, $BaCuO_2$ 12% and $Ba_2Cu_3O_6$ 48%, respectively. It can be seen that the samples prepared by melt process reduced the non-superconducting compound. However, the samples prepared by solid state reaction had a *c* lattice parameter longer than the samples prepared by the melt process method. The samples prepared by solid state reaction had a critical temperature higher than the samples prepared by melt process caused liquid phase [8] on the surface was non-superconducting compound [9].

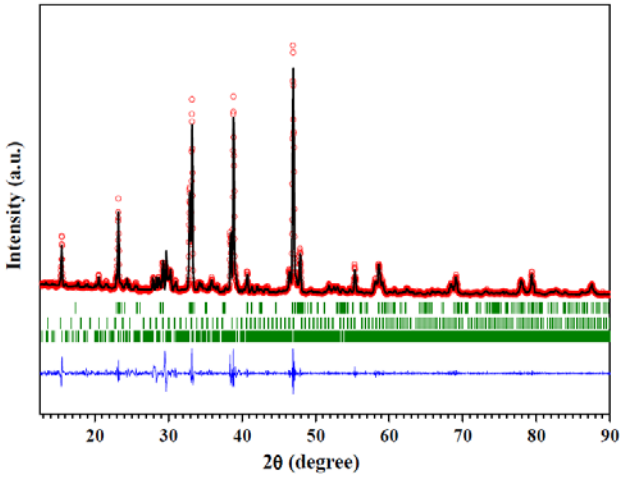


**Figure 4.** XRD pattern of melt Y5-8-13 superconductors.

The result of EDX mapping techniques is shown in figure 5. The results showed the area of the measurement. The samples consisted of the Y, Ba, Cu and O as represented in pink, red, blue and green color and without impurities, respectively. Grain size of the samples were homogenous, smooth and denser. The result of the elementary mapping shows that Y and O were inhomogeneously distributed while Ba and Cu distributed homogenously in Figure 6. show the EDX spectra of the samples with corresponding EDX mapping measurement.

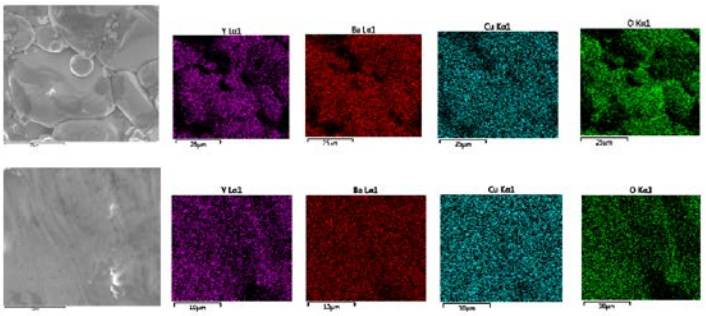

**Figure 5.** EDX mapping images of the melt Y5-8-13.

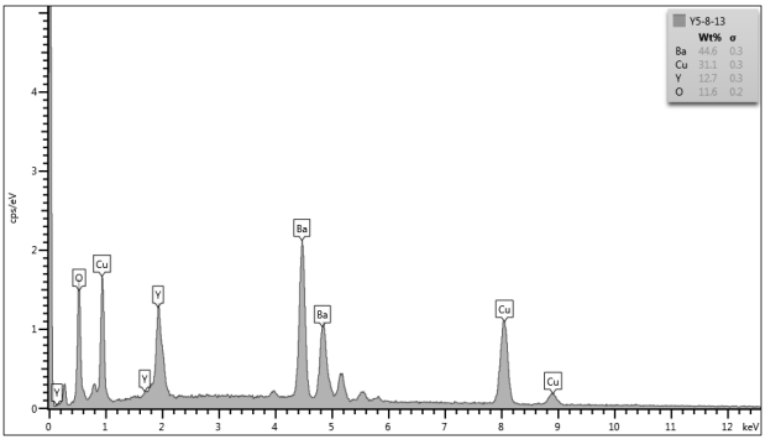


**Figure 6.** The EDX spectra of the melt Y5-8-13

Additionally, the melt process was widely used for synthesis of Y-based superconductors. There are other ways to synthesize this material such as the sol gel method [10], Liquid Phase Processing (LPP) [11] and Top Seed Melt Growth (TSMG) [12], etc. All of these approaches aim to improve the physical properties such as the ability to transfer the critical current density [13] and critical magnetic field [14], and mechanical and structural properties [15, 16].

## 4. Conclusions

Y5-8-13 superconductors were synthesized by the melt process. The four-probe method, XRD, EDX were used to investigate physical properties. The average critical temperature about was 91 K. The superconducting compound and non-superconducting compound were composite in the samples. The percentage of superconducting compound and non-superconducting compound were 30 % and 70%. The lattice parameter of the first compound was $a$=3.82912 A, $b$=3.88788 A and $c$=48.46370 A. The second compound separated in two types, 35% of Y211 ($Y_2BaCuO_5$) and 35% of $BaCuO_2$, respectively. The Y211 had lattice parameters $a$=7.21450 A, $b$=12.23320 A and $c$=5.62140 A and, $BaCuO_2$ was lattice parameter $a=b=c$=18.23560 A. The prepared samples reduced the non-superconducting compound and the surface was more homogenous, smoother and denser.

## 5. Acknowledgements

The authors thank the Research and Development Institute of Suratthani Rajabhat University, The faculty of Science and Technology for financial support.

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