Experimental Studies of the Effective Sintering Temperature on Electrical and Structural Properties of HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ Compound

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Abstract: The HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ specimens are manufactured via solid status interaction process at various temperature of sintering at (1073, 1123 and 1173) K. All the specimens in the immediately studied were submitted to common constructional description by X-ray diffraction. The X-ray diffraction data combined from different samples agree with (Hg - 1223) stages, then the investigations indicated that tetragonal construct for all specimens with an increasing in the dimension c to 15.87 Å and reducing the mass density ($\rho_m$) to 1.481 g/cm$^3$ at 1173 K. The four - sensor procedure was achieved to detection the transition temperature (Tc) , then it raised from 116 K to 121 K with increasing sintering temperature from 1073 K to 1123 K and decreased to 109 K at 1173 K.

Keywords: Sintering temperature, HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$.

1. Introduction

The mercury - based superconducting cuprates which is known as the compound HgCa$_n$Cu$_n$Ba$_2$O$_{2n+2}\delta$ (where n = 1 to 8 : n represent Cu - O levels), its considered as the generality important symmetrical groups from superconducting cuprates compounds that possess elevated transition temperature (Tc) displayed via this groups [1, 2]. Transition temperature (Tc) was 94 K for (Hg -1201, n = 1), while it was 127 K for (Hg -1212, n = 2) and 135 K for (Hg -1223, n = 3). Moreover the transition temperature (Tc) was raised up between 150 K to 160 K at elevated compression [3].

The crystal structure for all superconducting phases for the compound HgCa$_n$Cu$_n$Ba$_2$O$_{2n+2}\delta$ (where n = 1 to 8) is tetragonal included perovskite levels [4]. The mercury - based superconducting cuprates has structure most likely similar to structure of Tl and Cu - based superconducting cuprates with a single Tl - O layer but a substantial variation is that , the Hg - O$_8$ layers have more oxygen deficient, while the Tl - O layers have extremely few oxygen voids. A low binding for oxygen atoms with Hg atoms and the occupation of oxygen atoms is possible to vary above enormous domain depending on the manufacturing; it was shown for different sections of the mercury symmetrical group [5]. Where Copper - Oxygen levels are existent, which are accountable for the superconductivity at elevated transition temperature [6].
Unfortunately, the existence of CO$_2$ and wetness is still one of the problems concerning the phase stability. Some records exhibit that the cation substitution support stage construction and characteristics of superconductivity for Hg - 1223. The doping via high - valence kind Cu, Cd, Zn, Ti, Ag, Sb or other elements are able to improve the critical density of current and stage construction of ( Hg - 1223 ) [ 7 - 13 ].

Electrical resistivity ( \( \rho \) ) is very important characteristic of material, which through it can be determine the critical temperature of a superconductor. In this research many important measurements to study the constructional and electrical characteristics of bulk HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$, such as X - radiation deviation and the resistance of electrical. Samples are prepared using solid status interaction process at various temperature of sintering ( 1073, 1123 and 1173 ) K.

2. Experimental

A Hg-Ba$_2$Ca$_2$Cu$_3$O specimen has manufactured from the stoichiometric quantities of cationic proportion of Hg : Ba : Ca : Cu = 1 : 2 : 2 : 3 via solid status interaction process. The blend without Hg was stirred in 2 - methylethanol and allowed for perfect blending by magnetic stirrer for about 3 hours, then the solution has dried and evaporated at 673 K for one hour until the blend powder was obtained. The blend powder was calcined at 973 K for 12 hours, then the calcined powder was taken and blended with the varying concentration of mercury oxide.

The powder of HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ compound was grinded for 3 hours, then it was separated into three equivalent parts ( A, B and C) each one weighing 6 grams and these parts were pressed into pellets to obtain three samples. The sample A sintered at 1073 K, the samples B and C sintered at 1123 K and 1173 K respectively, then each sample cooled to 733 K for 5 hrs. in an oxygen atmosphere for annealing.

The crystal structure for the prepared samples of the HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ compound were achieved using X - radiation diffractometer kind ( Shemadze ). It possess the following characteristics: provenance is copper K\( \alpha \), current 30 mA, electric potential difference 40 kV, length of wave 1.5405 Å, deviation angle ( 10 - 80 ) deg, speed of scan 8 deg / min. The computer platform was employed to determine the unit cell constants (a, b and c) and its based on complete prof collection toolbar [ 8, 9, 14 ].

The standard four - sensor procedure was employed for calculate temperature of transition with a temperature ranging between 77 K to 300 K by measuring the temperature dependent resistance \( \rho \) ( T ) [ 15 ]. The sample has been installed in a cooling system linked to a rotating discharger in order to get compression of 6 x 10$^{-2}$ mbar in the cooling system and as well it linked to a detector of modern thermocounter near the sample location. Fine wires of Cu joined to the sample by Ag glue served as electrical points, then source of D.C with 20 mA was provided to the sample. The electric potential difference decline was recorded by voltagecounter with very high allergy. The specific resistance ( \( \rho \) ) was recorded from the equation [ 16 ]:

\[
\rho = \frac{V \omega h}{I d}
\]

Where \( V \): the electric potential difference decline through the electrical points, \( I \): the current that penetrates the specimen, \( \omega \): width of the specimen, \( h \): height of the specimen, \( d \): the actual dimension between the electrical points.

3. Results and Discussion

The peaks of X - radiation deviation shapes versus with the deviation angle for the samples ( A, B, C ) of HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ compound that were manufactured via solid status interaction process at various temperature of sintering ( 1073, 1123 and 1173 ) K are shown in figure 1. The XRD data taken from various samples agree with ( Hg - 1223 ) stages and the investigations indicated that tetragonal construct [ 11, 16 ].
From figure 1 discovered that increase the sintering temperature create variation in high phases. Schedule 1 indicate to variation in a and c lattice constants, the cause is due to the high temperature of sintering and injection Cu - O layer in the structure make extension at the basis construct of mercury oxide which produce an increasing in the dimension of a and c. Analogous consequences were obtained by other authors [17, 18].

Figure 1. X-ray radiation deviation shapes of HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ specimens at various sintering temperature (1073, 1123 and 1173) K.

The high sintering temperature causes an increasing in the dimension of a and c as well as volume the unit cell, while the mass density ($\rho_M$) reducing as offering in the Schedule 1 and figure 2. Analogous consequences were obtained by other authors [17].

Figure 2. Mass density ($\rho_M$) dependence of various sintering temperature (1073, 1123 and 1173) K for HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ specimens.
Figure 3 display the electrical resistivity against temperature of HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ component with various temperature of sintering (1073, 1123 and 1173 K), where that the transition temperature is determined from this figure. For specimen which sintered at 1073 K, the transition temperature at onset the transition $T_s$ (onset) from natural status to the exceptional case of electrical conductivity status (beginning of collapse for resistivity) was realized at 131 K and temperature of the transition $T_s$ (offset) when resistivity reaches to zero was realized at 116 K. The variation in width of the transition ($\Delta T = 15$ K). When the sintering temperature increase to 1123 K, also the curve of electrical resistivity exhibit a variation from natural status to the exceptional case of electrical conductivity status, while the value of $T_s$ (onset) became 134 K, $T_s$ (offset) 121 K and the variation in width of the transition ($\Delta T = 13$ K), while they decrease to 119 K and 109 K when the temperature of sintering increase to 1173 K as display in the figure 4.

This demeanor mentions that the perfect reaction and phase construction (Hg - 1223) with the greatest possible ratio for the HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ compound occurs when the sintering temperature is 1123 K in which the transition temperature is 121 K. Then decrease the transition temperature to 109 k after increase the sintering temperature to 1173 k perhaps because of increasing of the structural defects as mentioned in ref. [8].

![Graph](image1)

**Figure 3.** Temperature dependence of resistivity for HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ specimens with various sintering temperature (1073, 1123 and 1173 K).

![Graph](image2)

**Figure 4.** Values of transition temperature dependence of sintering temperature for HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ specimens.
**Schedule 1.** Values of transition temperature, lattice constants (a, c) and mass density ($\rho_M$) for the HgBa$_2$Ca$_2$Cu$_3$O$_{y+\delta}$ specimens with various sintering temperature (1073, 1123 and 1173) K.

| Sintering Temperature (K) | $T_s$ (offset) (K) | $T_s$ (onset) (K) | a (Å) | c (Å) | $\rho_M$ (g/cm$^3$) |
|---------------------------|--------------------|-------------------|-------|-------|------------------|
| 1073                      | 116                | 131               | 3.79  | 15.32 | 1.621            |
| 1123                      | 121                | 134               | 3.86  | 15.46 | 1.617            |
| 1173                      | 109                | 119               | 3.39  | 15.87 | 1.481            |

4. Inferences

In this immediate research, we have perfectly manufactured HgBa$_2$Ca$_2$Cu$_3$O$_{y+\delta}$ specimens at different temperature of sintering (1073, 1123 and 1173) K via solid status interaction method. The X-ray radiation diffractometer was used to find out the crystal structure for the prepared specimens of the HgBa$_2$Ca$_2$Cu$_3$O$_{y+\delta}$ compound. It was tetragonal for all the specimens with increasing the dimension of c from (15.32 to 15.87) Å and reducing the mass density ($\rho_M$) from (1.621 to 1.481) g/cm$^3$ at increasing sintering temperature from (1073 to 1173) K.

The four-sensor procedure was employed to determine the temperature of transition ($T_c$) and it was discovered that $T_c$ increase from 116 K to 121 K with increasing sintering temperature from 1073 K to 1123 K and then decrease to 109 K at 1173 K.

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