# The Fabrication and Characterization Superconducting of Compound $Hg_{2-x}Tl_x Sr_2Ca_2Cu_3O_{10+\delta}$

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# Abstract

The samples of high temperature superconductors of the type (2223), (Hg  $_{2-x}$  Tl<sub>x</sub> Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+6</sub>) have been prepared through the method of solid state reaction process. The study deals with the effect of substitution of Tl in Hg with different rates (x = 0.0 - 0.4) at variable sintering temperature, sintering time and annealing at 850  $^{0}$ C for 12 hours, at average heating 2  $^{0}$ C/min. The best T<sub>c</sub> value obtained for the compound was x = 0.2.

The results of x-ray test for the samples show that the Crystal structure is atetragonal structure. The samples contain a high ratio of Hg-2223 superconducting phase. The substitution of Tl with Hg leads to the increase of the grain size of superconducting phase .

Key words : Superconductor, Critical Temperature .tetragonal structure

## Introduction

Superconductivity is a Physical phenomenon which is customarily described without grundging epithets . Indeed , its nature had remained unclear for many years . To discover the mechanism of this phenomenon , several decades of efforts of many Physicists were needed . But even now , after the discovery in 1986 and 1987 of the so – called high – temperature superconductors , superconductivity is still under a veil of mystery – some key question have yet to be answered . But to approach them , we first have to trace the path traversed . we shall tell you what superconductivity is , how it was discovered , what its main properties are , where superconductors are applied now and haw they are most tickly to be widely used in future .

Valldor et.al. (2000)[1] studied the  $(Tl,Hg)_2(Ba,Sr)_2(Sr,Ca)_2Cu_2O_{8+}$  superconductor system. They found that  $T_c$  varies within (45-100) K with a decreasing formal oxidation state of Cu in the superconducting plane. On the other hand it is found that the interplanar distances increase proportionally with the critical temperatures. Indeed this distance increases with the Ba/Sr ratios on the larger metal.

Bhattacharya et.al. (2001)[2] reported investigation on converting electron deposited Tl-oxide films to Hg-1212 using the cation exchange process. They found that the superconducting transition temperature for the "non substitution" films improved from 97 to 120 after the cation exchange process.

#### **Experimental part**

Appropriate amounts of high purity  $Hg_2O$ ,  $Tl_2O_3$ ,  $Sr(NO_3)_2$ , CaO and CuO powders have been weighed stoichiometrically . The total weight of the compounds was taken as follows :

 $\begin{array}{ll} W_1(Hg_2O) = (2\text{-}x/2)[2\text{*}200.5\text{+}15.999] = 416.99 \ gm \\ W_1(Tl_2O_3) = (x/2)[2\text{*}204.37\text{+}3\text{*}15.999] = 456.74 \\ W_3(Sr(NO_3)_2) &= 2[87.62\text{+}2(14.007\text{+}3\text{*}15.999)] \\ = 423.26 \end{array}$ 

 $W_4(CaO) = 2[40.08+15.999] = 112.14$ 

 $W_5(CuO) = 3[63.546+15.999] = 238.59$ 

Any different types of superconducting systems according to their nominal composition with x=0 to x = 0.4 for both Hg and Tl were prepared by a two step method and as follows :

1. Measuring the weight of each reactant by using a sensitive balance with 4-digit type (Mettler H35AR).

2. Mixing and grinding the powders  $(Tl_2O_3, Hg_2O)$ and  $(Sr(NO_3)_2$  CaO, CuO) together by using a gate morter, the mixture homogenization took place by adding a sufficient quantity of 2-propanol to make think slurry. This process was repeated several times to produce a homogenous, soft and as fine as powder particles.

3. Drying the mixture in oven at  $80^{\circ}$ C for 60 min.

4. Measuring the weight of the dried mixture  $(W_1)$ , and the it in an alumina crucible.

5. Putting the mixture in a tube furnace that has programmable controller type (Eurothrem 818) for calcinations in air, which is the best treatment to remove (CO<sub>2</sub> and NO<sub>3</sub>) gases from the mixture. The powder was heated to temperature at  $850^{\circ}$ C for 24 hours with a heating rate of  $2^{\circ}$ C/min, then cooled to room temperature by the same rate.

6. Measuring the weight of the mixture after calcenation  $(W_2)$ , the color of it was black. If the difference in the sample weight before and after the calcinations process  $(W_2-W_1)$  is less than the theoretical value of gases, as it is always the case, then the steps 2,3,4,5 should be repeated again for two or three times to remove the whole gases from the mixture.

7. Adding Hg<sub>2</sub>O or Tl<sub>2</sub>O<sub>3</sub> to the mixture Sr-Ca-Cu-O and grinding them in a gate morter for about 30 min to obtain a very fine and optimum homogenous powder. The mixtures were pressed into a pellet of (0.2-0.3)cm in thickness and 1.3 cm in diameter, under a pressure of about 8 ton/cm<sup>2</sup> by using hydraulic press type (specac).

8. Sintering the pellets in air atmosphere of  $850^{\circ}$ C for 12h with a heating rate  $2^{\circ}$ C/min in order to make the particles of the materials ionized together and gradually reduce the volume of pore spaces between them. Then the pellets were cooled to room temperature by the same rate of heating.

9. Regrinding, repressing and resintering the sintered pellets were reground, repressed and resintered in air at the same rang of temperature for further 12h.

10. In this work, three sets of samples were produced for each superconducting compound. The first set of samples was cooled to room temperature in the furnace with a cooling rate  $2^{0}$ C/min, the second set of samples was removed from the furnace and quenched in air, while the third set of samples was sintered for 12h in flow O<sub>2</sub>.

#### **Results and Discussion**

We will present our results and discuss their mean features. This will include the x-ray diffraction results and analysis (XRD).

#### X-Ray Diffraction (XRD):

The powder x-ray diffraction method has been used for structural characterization and to determine the lattice parameters of the samples processed and used in this study , by using computer controlled diffractometer.

#### **X-Ray Diffraction Analysis :**

X-ray diffraction type (Phillips), having the following features, was used to examine the structure of the prepared samples.

Source : Cu K $\alpha$ 

Voltage: 40 Kv

Current: 20 mA

Wavelength :  $1.5405 \text{ A}^{0}$ 

A computer program [3] was workedout to calculate the lattice parameters a,b,c. This program is based on Cohen's least square method[4].

The XRD technique has been used in our study to calculate the lattice parameters and the size of unit cell. In this technique an electromagnetic radiation (beam of X-ray) of high energy and short wavelength were used.

The wavelength is of the order of the atomic spacing for solids-incident on a solid crystalline material. A part of beam will be scattered due to the electrons associated with each atom or ions that lies within the path of the beam. If we consider an X-ray of wavelength  $\lambda$  is incident at an angle  $\theta$  on two parallel planes of atoms that are separated by interplanar spacing d<sub>hkl</sub>, and then get constructive interference if Bragg's law satisfied:

 $n \lambda = 2d_{hkl}sin(\theta)$  -----(1)

Where n is the order of reflection,  $\lambda$  is the wavelength of incident X-ray, d is the distance between two parallel planes of atoms(interplanar spacing d<sub>hkl</sub>),  $\Theta$ is the angle between the plane and incident beam of X-ray. In our study,  $\lambda$  is fixed by using cooper or cobalt source, for cooper source  $\lambda$  is equal 1.54056 A<sup>0</sup> and n=1. We determine the angle  $\theta$  from the peak position in diffraction pattern then we calculate d using equation (1). Each value of d associated with a set of Miller indices (h k l). The lattice parameters(a,b,c) are determined using the following equation (2)[5]:

 $1/d^2 = h^2/a^2 + k^2/b^2 + l^2/c^2$  -----(2)

A powder of these samples has been used. The data obtained by using computer controller diffractometer. Our results for the type 2223, Hg  $_{2-x}$  Tl<sub>x</sub> Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\delta$ </sub> Systems have been prepared by solid state reaction is shown in figures(1-5).

#### Intensity C/s



Fig.(1): X-ray diffraction patterns of Hg  $_2$  Tl\_0Sr\_2Ca\_2Cu\_3O\_{10+\delta} System

Intensity C/s



Fig.(2): X-ray diffraction patterns of Hg  $_{1.9}$  Tl\_{0.1}Sr\_2Ca\_2Cu\_3O\_{10+\delta} System

Intensity C/s



Fig.(3): X-ray diffraction patterns of Hg  $_{1.8}$  Tl\_{0.2}Sr\_2Ca\_2Cu\_3O\_{10+\delta} System

Intensity C/s











By comparing the peaks positions in the X-ray diffraction and we calculate the interplanar spacing (d-values) at each peak in the spectra by using equation (1):

Where n=1 and  $\lambda = 1.54056$  for copper source. For each value of d obtained using equation (1), there are a set of Miller indices (h,k,l), then we calculate the lattice parameter (a,b,c) by using equation (2):

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Samples	$\mathbf{a} = \mathbf{b} (\mathbf{A}^0)$	$c(A^0)$	a/b=1	c> a,b	$\mathbf{d}(\mathbf{A}^{0})$
$Hg_{2}Tl_{0.0}Sr_{2}Ca_{2}Cu_{3}O_{10+\delta}$	4.167	35.433	a/b=1	c> a,b	2.9
$Hg_{1.9} Tl_{0.1} Sr_2 Ca_2 Cu_3 O_{10+\delta}$	4.113	35.654	a/b=1	c> a,b	2.8
$Hg_{1.8}Tl_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$	4.103	36.764	a/b=1	c> a,b	2.7
$Hg_{1.7} Tl_{0.3} Sr_2 Ca_2 Cu_3 O_{10+\delta}$	4.211	34.543	a/b=1	c> a,b	3.0
$Hg_{1.6}Tl_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$	4.258	33.655	a/b=1	c> a,b	3.1

Table (1) The value of parameters a,b,c with different subistituation

The x-ray data of all superconductor samples showed a tetragonal structure with a high ratio of Hg-2223 superconducting phase.

Fig (6) show the variation of resistivity with temperature for  $Hg_{2-x} Tl_x Sr_2Ca_2Cu_3O_{10+\delta}$  system with

different x (x=0 to x= 0.4). From these figure we can see the critical transition temperature. The values of the critical transition temperature  $T_c$  of these samples are listed in Table (2) :

Table (2) The The values of the critical transition temperature  $T_c$  of these samples

Samples	critical transition temperature T <sub>c</sub>
$Hg_{2}Tl_{0.0}Sr_{2}Ca_{2}Cu_{3}O_{10+\delta}$	123 K
$Hg_{1.9} Tl_{0.1} Sr_2 Ca_2 Cu_3 O_{10+\delta}$	124 K
$Hg_{1.8} Tl_{0.2} Sr_2 Ca_2 Cu_3 O_{10+\delta}$	142 K
$Hg_{1.7} Tl_{0.3} Sr_2 Ca_2 Cu_3 O_{10+\delta}$	135K
$Hg_{1.6}Tl_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$	131 K



Fig(6): Temperature dependence of resistivity for Hg  $_{2-x}$  Tl<sub>x</sub> Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\delta$ </sub> system with different x (x=0 to x= 0.4).

#### **Conclusions :**

The doping of Tl in Hg for the compound Hg  $_{2-x}$  Tl<sub>x</sub> Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\delta$ </sub> with (x = 0.0 - 0.4) has exhibited a maximum value of T<sub>c</sub> which found to be (123,124,142,135,131) K. The best T<sub>c</sub> value obtained for the compound is x = 0.2.

# **Suggestions for Future :**

1-Studying other important parameters , such as the critical current , critical field.

2 – Preparing HTSC as a thin film by a pulsed laser deposition technique.

3 – Studing the effect of proton and ion irradiation on the physical properties and superconductivity of the prepared samples.

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# تحضير ودراسة خواص المركب Hg<sub>2-x</sub> Tl<sub>x</sub> Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+6</sub> فائق التوصيل

# نهاد على شفيق

قسم الفيزياء ، كلية التربية ، جامعة تكريت ، تكريت ، العراق ( تاريخ الاستلام: ٢ / ١ / ٢٠١١ ---- تاريخ القبول: ٢٨ / ٦ / ٢٠١١ )

# الملخص:

تم تحضير عينات فائقة التوصيل عند درجات الحرارة العالية من نوع (٢٢٢٣)  $Hg_{2-x} Tl_x Sr_2 Ca_2 Cu_3 O_{10+\delta}$  (٢٢٢٣) تم تحضير عينات فائقة التوصيل عند درجات الحرارة العالية من نوع (٢٢٣)  $Hg_{2-x} Tl_x Sr_2 Ca_2 Cu_3 O_{10+\delta}$  الصلبة. تم دراسة تأثير تعويض Tl في Hg بنسب مختلفة (c = 0.0 - 0.4) عند درجات تلبيد مختلفة، زمن مختلف وتلدين عند 850 لمدة (r = 0.0 - 0.4) عند درجات تلبيد مختلفة، زمن مختلف وتلدين عند c = 0.4 لمدة الصلبة. تم دراسة تأثير تعويض Tl في  $r_c$  افضل قيمة  $T_c$  تم الحصول عليها للمركب عند c = 0.2

نتائج فحص الاشعة السينية للعينات بينت ان التركيب البلوري هو من النوع الرباعي القائم، والعينات تحتوي على نسبة عالية من الطور - Hg 2223 وكذلك ادّى تعويض TI بـ Hg الى زيادة حجم حبيبات طور التوصيل الفائق.