https://doi.org/10.30526/31.3.2024



Vol. 31 (3) 2018

# Synthesis and Study the Structural and Electrical and Mechanical Properties of High Temperature Superconductor Tl<sub>0.5</sub>Pb<sub>0.5</sub>Ba<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n-x</sub>Ni<sub>x</sub>O<sub>2n+3-δ</sub> Substituted with Nickel Oxide for n=3

## Laheeb A. Mohammed

Kareem A. Jasim Department of Physics, College of Education for Pure Science Ibn-Al-Haitham, University of Baghdad, Baghdad, Iraq Laheebfrahmed@yahoo.com

Article history: Received 21 May 2018, Accepted 4 September 2018, Published December 2018

## Abstract

The effect of nickel oxide substitution on the pure phases superconductor  $Tl_{0.5}Pb_{0.5}Ba_2Ca_{n-1}Cu_{n-x}Ni_xO_{2n+3-\delta}$  (n=3) where x = (0, 0.2, 0.4, 0.6, 0.8 and 1.0) is studied in this research. The specimens in this work were prepared with used procedure of solid state reaction with sintering temperature 850°C for 24 h. We used technical (4-prob) to calculated and the critical temperature T<sub>c</sub>. The results of the XRD diffraction analysis showed that the structure for pure and doped phases was tetragonal with phases high-T<sub>c</sub> phase (1223), (1212) and low-T<sub>c</sub> phase (1202) and add to the presence of some impure phase. It was noted the value a=b,c the parameter of the lattice increment with the increment of Ni content. The increment of (NiO) concentration effects electrical resistivity, dielectric constant and the hardness.

Keywords: Superconductors, critical temperature, solid state reaction, dielectric constant.

## 1. Introduction

Superconductivity, which is explained as a phase transition of electrons, is a phenomenon by which a conductor loss all resistance to electric current under specific conditions and exhibits complete diamagnetism.

Superconductivity is the property of completely electrical resistance lack in some materials when they cooled below a certain temperature called critical or transition temperature (T<sub>c</sub>) which varies from material to another [1,2]. The high temperature superconductivity of cuprates was discovered in1986. Tl-Ba-Ca-Cu-O this family of superconductors has been discovered in1988 by Sheng and Hermann is the best among all other cuprates due to their high transition temperature, transport critical current densities and low microwave surface Resistances. TBCCO materials have perovskite like Ba<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>2n+1</sub> layers and tetragonal crystal structures There are two Tl-based superconducting families, TBCCO and TSCCO. The first series of the Tl-based superconductor containing one Tl- O layer has the general formula TlBa<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>2n+3</sub> with n=3. In this paper was focused on the study the effect the partial substitution of copper with nickel oxide on the structural, electrical and mechanical properties of the superconducting [3].

https://doi.org/10.30526/31.3.2024

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## 2. Experimental

The  $Tl_{0.5}Pb_{0.5} Ba_2Ca_2Cu_{3-x}Ni_xO_{8+\delta}$  with (x=0,0.2,0.4,0.6,0.8 and 1.0) high critical temperature superconducting (HTS) was synthesized by solid state reaction process, Using the required amounts of pure powders materials high purity oxides (99.99%) of Tl<sub>2</sub>O<sub>3</sub>.PbO,NiO, BaO,CaO and CuO, and in Commensurate with the molecular weights, accordingly for these chemical formulas:

 $[0.25\text{Tl}_2\text{O}_3+0.5\text{PbO}]+2\text{BaO}+2\text{CaO}+(3-x)\text{CuO}+x\text{NiO} \rightarrow \text{Tl}_{0.5}\text{Pb}_{0.5}Ba_2Ca_2Cu_{3-x}Ni_xO_{8+\delta}.$ 

The reactants were measured by using a sensitive balance whose sensitivity order  $(10^{-4})$  g. The reactants were mixed jointly by a gate mortar with addition of isopropanol (C<sub>2</sub>H<sub>3</sub>O<sub>5</sub>) was to homogenize the mixture dry slurry during the grinding process for nearly (from 40 to 60) minute. The mixture was put in an alumina crucible and dehydrated for in the oven at 120°C. The mixture was compressed into the disc-shape as pellets with diameter 1.5 cm and (0.2 0.3)cm thick; by hydraulic compress under a (7 ton/cm<sup>2</sup>) pressure. Disks putted in a furnace and sintered at 850°C for 24 hours with the rate of (5 °C/min) and then cooled to the room temperature by the same rate. Structure of crystal such as phase of crystalline, the polycrystalline, amorphous, grain size, and parameter of lattice of all specimens prepared were examined by XRD technique system (SHIMADZU Japan XRD 600) by records the intensity in the range of Bragg's angle  $\theta$  from (20-80), Cu (K $\alpha$ ) radiation source of wavelength ( $\lambda$ =1.5405 Å) was employed with generator setting of current 20 mA and voltage 40 kV. The parameter of unit cell was determined using by the software of the check cell computer. The morphology of surface for these specimens observed from the ((AFM)) technique, through using SPM model AA3000 contact mode spectrometer, supplied by Angstrom Advanced Inc [4]. Four probe method at temperature range (77-300) K was used to measure the resistivity( $\rho$ ) [5]. Critical temperature (T<sub>c</sub>) calculated using the relation:  $\rho = (R * A)/L$ (1)

Where R is electric resistance, A is area of specimens and L is length of specimens.

The mechanical properties were checked by Vickers MicroHardness, measurements of hardness for the specimens were carried out on the polished surface of the tested specimens at room temperature. The test Vickers hardness used the square-base diamond pyramid as the indenter and the angle of (136) degrees between faces opposite subjected the applied load, F, was 2.940 N. The full load is normally applied for 15sec [6,7].

 $Hv = 1.8544 (F/d^2)$ 

Where: F : the amount of inflicted force and d: arithmetic mean of the two diagonals in mm

The Young modulus E of superconductors is related to the Vickers microhardness by the relation [7]:

$$E = 81.96 \text{ Hv}$$
 (3)

The yield strength Y is related to the hardness by the relation [7]:

Y = Hv / 3

The dielectric constant was determined using (HF LCR meter) 6500p Series, Uk, frequency range from (20Hz-120MHz). measurements the dielectric properties that dependent of frequency using a device of LCR meter at frequencies from (50Hz-5 MHz), Where it has been measured the conductance (G), the capacitance (C), dialectic constant ( $\dot{\epsilon}$ ), The loss of dielectric ( tan $\delta$  ) and conductivity ac (  $\sigma_{a.c}$  ) for all specimens that determined by the Equations (5, 6, & 7) [8, 9, 10]:



" $\varepsilon_r$ " dielectric loss factor, **d** is the thickness of the pellet, **A** is the area of the electrode,  $\omega = 2\pi f$  is the angular frequency,  $\varepsilon_0$  is the permittivity of free space =  $8.85*10^{-12}$  F/m.

## **3.Results and Discussion**

#### **3.1. Structural Properties**

The X-ray diffraction patterns results of pure and substituted Tl-based superconductors are shown in **Figure 1.** for (n=3). The main phases in these specimens are high Tc phase H (1223), M phase (1212) and low Tc phase L (1201) and amounts impurities



Figure 1. XRD patterns for the specimens of  $Tl_{0.5}Pb_{0.5}Ba_2Ca_2Cu_{x-3}Ni_xO_{9-\delta}$ when x=(0, 0.2, 0.4, 0.6, 0.8 and 1.0).

As for the parameters of the crystal lattice a, b, c and c/a, the results as shown in the **Table 1**. shows that for all samples the crystal structure is tetragonal. The c-axis lattice constant increase with the increase of NiO ratio, Maybe due to some strains and variation of  $O_{\delta}$  oxygen contents after NiO nanoparticles addition. These results were almost identical to those reported in reference [11].

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<b>Table 1.</b> Values of lattice parameter, c/a, pm for the sample $Tl_{0.5}Pb_{0.5}Ba_2Ca_2Cu_{x-3}Ni_xO_{9-\delta}$ .	
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x	a=b (A°)	c(A°)	c/a ratio	v(A°)³	dm(gm/cm ³)	V ph(1223) %	V ph(1212)%	V ph(1201) %	Vp impurities %
0	3.8135	15.2769	4.006005	222.1686	7.454971	74.9457	10.5206	8.7852	5.6399
0.2	3.7326	15.3323	4.107673	213.6142	7.745964	77.5539	11.8944	8.1534	8.4412
0.4	3.8221	15.4090	4.031553	225.1016	7.343511	76.0471	110852	9.07441	10.3266
0.6	3.8299	15.5589	4.062482	228.22	7.221973	72.619	16.522	5.114	5.743
0.8	3.8380	15.7034	4.091558	231.3149	7.132317	70.1117	8.8427	7.9448	87825
1.0	3.8379	15.8110	4.119701	232.8878	7.091071	83.8009	10.1357	5.8823	9.0497

 $\begin{array}{l} \textbf{Table 2.Transition temperature } T_{c(onset)}, (T_{c(offset)}, transition width (\Delta T_c), oxygen content ( \delta ) Vicker's Hardness \\ H_{v}, Young modulus (E) and Yield strength (Y) for Hg_{0.5}Pb_{0.5}Ba_{2}Ca_{2}Cu_{3-x}Ni_{x}O_{8+\delta}. \end{array}$ 

x	T <sub>C(Off)</sub> (K)	T <sub>C(On)</sub> (K)	ΔTc (K)	T <sub>C(mid)</sub> (K)	Eg (eV)	δ	Hv	Е	Y
0	113	124	11	118.5	0.0360	0.06190	189	15490.44	63
0.2	114	125	11	119.5	0.0363	0.0659	200	16392	66.6666
0.4	128	130	2	129	0.0392	0.2325	212	17375.52	70.6666
0.6	124	133	9	128.5	0.0390	0.0913	219	17949.24	73
0.8	132	135	3	133.5	0.0406	0.1116	225	18441	75
1.0	134	144	10	139	0.0421	0.1258	240	19670.4	80

The superconducting transition temperature  $T_c$  (offset) for pure and doped samples for (1223) phase is higher due to increasing Ca-O layer as compared with increasing Ni concentration. the critical temperature  $T_c$  (off) was increased from 118K to 138 K with increasing the Ni content at (x= 0.2 -1.0) which are agreement with reference [11,12]. We can be observed the absence resistivity of superconductor transition as shown in **Figure 2**. and from energy gap results at the **Table 2**. [13].



Figure 2. The resistivity dependence on Temperature for the sample  $Tl_{0.5}Pb_{0.5}Ba_2Ca_2Cu_{3-x}Ni_xO_{9-\delta}$  at x= 0.0, 0.2,0.4,0.6,0.8,1.0.

Also we can observe the decreasing in c/a ratio with increasing the Ni content. This may due to the ionic radii mutuality of Ni ion by Cu ion which takes a compensate places through the structure of the unit cell instead of the sites interstitial [14].

The results of **Hv**,**Y**,**E** show in general **Table 2.**, increasing in the hardness value with increasing the NiO content may due to the Nickel ferrites have interesting structural, electrical, mechanical, and magnetic properties ,it has been showing an increase in the value of hardness may due to without interference in the grain boundaries [15]

For superconductor  $Tl_{0.5}Pb_{0.5}Ba_2Ca_2Cu_{3-x}Ni_x O_{9+\delta}$  compound with (x = 0.1.0) the properties dielectric have been tested. The changes of dielectric constant are indicated in the **Figure 3**. where we observe a decrease in value with increasing frequency. Dipolar polarization at the lower frequency is dominant, but when increasing frequency, the oscillation of the field applied increment and the dipolar polarization becomes longer than of the time constant of the field applied. For this reason, a decrease was observed in  $\epsilon$ , carriers cannot track the frequency of the external voltage field applied at high frequencies, and the dielectric resonate becomes ineffective.



Figure 3. Variation of dielectric constant versus frequency.



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The imaginary part ( $\xi$ ) dependent on frequency variation of all specimens at room temperature that shown in **Figure 4**. All the attain specimens exhibit dielectric disperse where adecrease in is  $\xi$  that observed with increment in frequency. The phenomenon of dielectric disperse has been explained on the basis by the model of Maxwell–Wagner for dielectrics.



Figure 4. Variation of dielectric constant  $\varepsilon''$  versus frequency.

## 4. Conclusions

In the present work, we have prepared the high superconductor compound  $Tl_{0.5}Pb_{0.5}Ba_2Ca_{n-1}Cu_{n-x}Ni_xO_{2n+3-\delta}$  (n=3) with (x=0,0.2,0.4,0.6,0.8and 1) by method of solid state reaction, and study the partial substitution nickel oxide of copper for electrical and structure and mechanical properties. all specimen examining by XRD analyses shown tetragonal crystal structure and observed that c-parameter value increase with increase of Ni content. The best value of T<sub>c</sub> obtained for the x = 1.0 where T<sub>c</sub>= (139 K). As well that , increasing Ni concentration strengths interlayer bonding ascertain that by the increase in the Hv, E and Y values of  $Tl_{0.5}Pb_{0.5}Ba_2Ca_{n-1}Cu_{n-x}Ni_xO_{2n+3-\delta}$ .

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