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Effect of Partial Replacement of Mercury by Lead on Structural and Electrical Properties of $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ Superconductor

The physical properties of the $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ superconductors at x values of 0, 0.05, 0.1 and 0.2 were examined to the simultaneous partial substitution of lead (Pb) at the mercury site. The capsule's samples were prepared in three steps by solid-state reaction. All samples had tetragonal structure with two phases: a main phase of High-Tc Hg-1212 and a secondary phase of Low-Tc Hg-1201, as revealed by structural analysis. In addition, there were impurity phases like CaHgO_2 and CuO . The lattice parameter, P(Hole) concentration, and c/a values changed when the lead (Pb) concentration in the prepared $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ compound was increased from 0 to 0.2. The zero critical transition temperature for $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, were found to be 82, 86, 93 and 106 K, respectively, while the onset critical transition temperature was found to be 101, 103, 105 and 124 K respectively.

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1. Introduction

Today, the phenomenon of superconductivity is considered one of the most important phenomena in solid-state physics, because some metals and compounds that are characterized by this phenomenon have almost no specific resistance at a certain low temperature, or in other words, the electric current flows through them without any dissipation in its value. Thus, its conductivity becomes very superior. Historically, this phenomenon was discovered in 1911 by the Dutch physicist Heike Kamerlingh Onnes [1]. Another important characteristic that characterizes superconducting materials at their transformation temperature is their aversion to the magnetic field directed at them, that is, they reflect the magnetic field no matter how weak its intensity. When a material is chilled below a particular temperature [2], it exhibits perfect diamagnetic behavior, has zero resistance, and exhibits this feature macroscopically. Thus, the properties of these materials have opened the doors for scientists to exploit them in innovations and inventions of high efficiency that are involved in most Fields of science and technology [3]. Although the topic of superconductivity is one of the most promising in material science, it is difficult to enhance its features in novel materials and compounds. Many ideas and models have been developed to describe this behavior, but they are still insufficient to represent all superconductor materials [4]. Subsequently, the superconducting materials were classified into two types: the traditional type, which belongs to metal alloys and metallic elements,

with a transition temperature lower than that of liquid nitrogen, and the other type, ceramic compounds with a temperature higher than 77 K, which is based on cuprite. Including mercury compounds containing copper layers. In 1986, high-temperature superconductors-ceramic materials with a critical temperature higher than 90 K were discovered [5]. This discovery was significant because it made it feasible to employ liquid nitrogen for cooling, ushering in a new age for superconductors [6]. The discovery of these materials enabled numerous experiments and applications and research is still ongoing to find superconducting materials with higher critical temperatures to increase the number of applications. Cryogenic liquid nitrogen boils at 77 K degrees, and its cost is affordable [7].

The homoerotic series of mercury coppers in the Hg-Ba-Ca-Cu-O family has the chemical formula $\text{HgBa}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+2+\delta}$ (HBCCO). Research interest has been generated by the discovery of high-temperature superconductivity in $\text{HgBa}_2\text{CuO}_{4+\delta}$ (n=1) [8], the higher transition temperatures $T_c = 120$ K was found in $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$ (n=2) [9,10], and $T_c = 135$ K for the compound $\text{HgBa}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\delta}$ [11]. The latter phase still maintains the record for both a cuprite-superconductor with transition temperature $T_c(\text{onset})$ is 164 K at a semi-hydrostatic pressure of 31 GPa and for the highest critical temperature T_c of any superconductor at ambient pressure [12]. The mercury copper superconductor was manufactured with n layers of copper oxide, ranging from n= 1 to 7 [13]. It was shown that the transition temperature

climbs to a maximum at $n = 3$ and then drops after that at the value of $n > 3$.

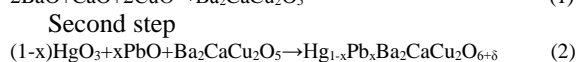
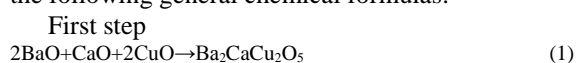
Mercury copper is difficult to produce and handle due to the very volatile and poisonous nature of mercury-based compounds, in contrast to its promising superconducting qualities. High-quality specimen manufacturing is necessary for both the prospective successful application as well as for the measurement of crucial intrinsic features [14]. Several teams completed this task successfully [15], mainly by using the partial replacement of some chemical elements of the compound with other elements of higher valiancy to obtain more gaps in the compound as well as increase the stability and transition temperature of O_2 [16-18].

There have been many studies investigating electrical transport characteristics in samples with various structural characteristics [19]. The characteristics of HBCCO samples reported by different papers varied greatly as a result of the particular preparation circumstances. It is yet unknown if this variability results from somewhat varied preparation circumstances in various laboratories or if it might also happen in purportedly the same production processes. For example, electrical resistivity in polymorphic samples of $HgBa_2CaCu_2O_6$ (Hg-1212) varied at room temperature by a factor of more than two across individual samples cut from the same ceramic but annealed for different durations [20]. Surprisingly, these samples' Hall effects were strikingly identical. On the other hand, following many annealing processes in various mercury and oxygen atmospheres, the resistance and Hall effect in Hg-1212 thin film both altered dramatically [21].

In this paper, the structural and electrical conductivity properties were studied for four superconductors $Hg_{1-x}Pb_xBa_2CaCu_2O_{6+\delta}$ with x values of 0, 0.05, 0.1 and 0.2, synthesized by the solid-state reaction method. We found that fundamental superconducting properties, such as critical temperature, and structural properties such as anisotropic lattice constants only show slight variation in samples with a difference in lead concentration.

2. Experimental Part

Four Samples of the superconducting mercury-based compounds ($HgBa_2CaCu_2O_{6+\delta}$, $Hg_{0.95}Pb_{0.05}Ba_2CaCu_2O_{6+\delta}$, $Hg_{0.9}Pb_{0.1}Ba_2CaCu_2O_{6+\delta}$, and $Hg_{0.8}Pb_{0.2}Ba_2CaCu_2O_{6+\delta}$) of phase 1212 were prepared, using appropriate weights of pure HgO, PbO, BaO, CaO, and CuO powders as starting materials. This was done using two steps according to the following general chemical formulas:



A sensitive balance was used to measure the weight of each reactant. The preparation process was

completed in three steps, in the first step, the precursor approach was used to synthesize the samples. Using an agate mortar, the powders of BaO, CaO and CuO were combined in the first stage. Enough 2-propane was added to the mixture to homogenize it and create a slurry while it was ground for 30 to 50 minutes. The mixture was dried in a 100°C oven. The combination was placed in a furnace with a programmed controller, a heat treatment used to get rid of the H_2O in the mixture. The nominal compositions $Ba_2Ca_2Cu_3O_4$ were obtained.

In the second step, to produce the combination $Hg_{1-x}Pb_xBa_2CaCu_2O_{6+\delta}$ with $x = 0.0, 0.05, 0.1$, and 0.2 , the precursor $Ba_2Ca_2Cu_3O_7$ was combined with HgO and PbO in an electric agate mortar for 40 minutes. The powdered particles are closely encapsulated inside separate vacuum capsules. The capsules were heated to a sintering temperature of (900°C) for six hours at a rate of 300°C per hour, and then they were cooled to room temperature at the same rate of heating. The capsules were taken out of the oven and each capsule was broken individually to extract its contents. The samples were then removed and ground in an agate mortar. Using a hydraulic press with a pressure of 5 tons per square centimeter, the powder was formed into pellets with a diameter of 1.5 cm and a thickness of 0.2-0.25 cm.

In the third step, the pellets were heated for 12 hours at a rate of 300°C per hour to the sintering temperature of $855-860^\circ\text{C}$ in oxygen (oxygen rate 0.2 L/min). Following annealing in the oxygen for two hours at 450°C , they were cooled to ambient temperature using the same rate of heating. Using x-ray diffraction (XRD) measurements (2θ in a range from 20 to 60°), the structure of the prepared samples was determined. The a and c lattice parameters were calculated using the computer program that was based on Cohen's least square approach [22].

The resistance vs. temperature (ρ - T) properties of these samples were evaluated using a four-probe method to consider the superconducting state. Using the technique described in reference [23], critical temperatures were measured.

The energy gap of the superconducting samples was then determined using the relationship shown below [24-26]

$$E_g = 3.53k_B T_c \quad (4)$$

$$p = 0.16 - \left[\frac{\left(1 - \frac{T_c}{T_{c(max)}}\right)}{82.6} \right]^{1/2} \quad (5)$$

where the critical temperature for the system is represented as $T_{c(max)}$ and p is the gap concentration

The critical temperature $T_{c(max)}$ for the prepared $Hg_{1-x}Pb_xBa_2CaCu_2O_{6+\delta}$ systems (with $x = 0, 0.05, 0.1, 0.2$) were determined. Since both copper and oxygen form a series of copper oxide layers that are inserted into the system's crystal structure and permit oxygen to enter and exit from within the composition, the structure of the $Hg_{1-x}Pb_xBa_2CaCu_2O_{6+\delta}$ superconducting ceramic system can be thought of as

an electrically active mass. Because superconductivity requires a precise concentration of cavities, the crystalline structure offers a way to achieve this.

3. Results and Discussion

In this section, the crystalline properties of four samples represented by the compounds $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ will be studied using XRD patterns to determine the effect of partial replacement of mercury with lead on experimental samples that were manufactured using solid-state reaction technology. The XRD analysis is shown in Fig. (1).

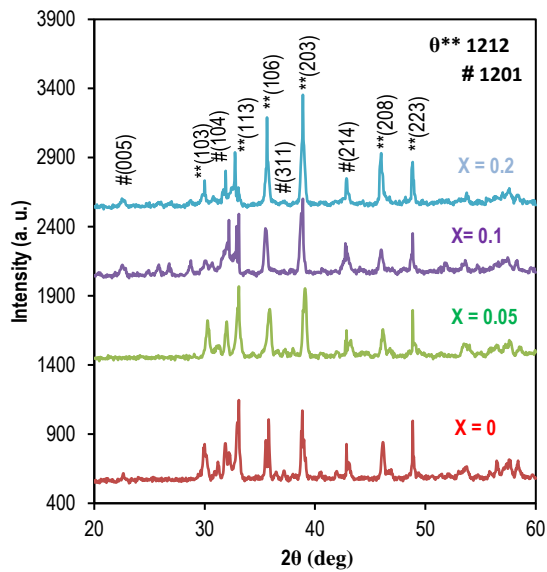


Fig. (1) XRD patterns of the Hg-1212 $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ samples

It can be seen from this figure that all samples are dominated by the Hg-1212 phase, as shown by calculations of the crystal lattice parameters a , b and c , with very little Hg-1201 and some pollutant phases remaining. As the lead content in the samples increased, the Hg-1212 phase increased with the direction of the c -axis perpendicular to the substrate surface, as shown in the peaks (**). The Hg-1212 phase is created by inserting a layer of calcium oxide into the Hg-1201(#) phase, which turns into the Hg-1212 phase. This result indicates that the absence of lead addition to the sample makes it difficult to increase the Hg-1212 phase. However, if Pb^{2+} is used instead of Hg^{2+} sites in general, followed by the same conditions. The increase in lead concentration in the samples led to the formation of the Hg-1223 phase and the eventual extinction of the Hg-1201 phase, and this was observed by calculating the volume fraction for each sample. The induction increased from 77% in the lead-free sample ($Q=0$) to 88%. By adding a

lead concentration of 0.2 as shown in table (1), which shows that both the lattice constants a and c vary when the lead concentration changes.

Figure (2) displays a plot of the normalized resistivity vs. temperature behavior of the superconductor samples $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ with different lead (Pb) concentrations (both doped and undoped). All of the samples' normal-state resistivity had metal-like characteristics about temperature. It has been demonstrated that the critical temperature T_c varies when lead (Pb) is added. Figure (2) shows two different systems. Above the beginning temperature and equivalent to the critical temperature T_c , the normal state that displays metallic characteristics is shown. This is the high-temperature portion when the normal resistance is seen to be linear and to have a slight decrease from room temperature to a certain temperature above the critical temperature T_c (onset), it was found that the critical transition temperatures T_c (onset) = 101, 103, 105 and 124 K with lead concentration = 0, 0.05, 0.1 and 0.2, respectively. The second section is the precise region where the electrical resistance exhibits a fast decline until it reaches zero with the assistance of the oscillation of copper pairs in conduction as T_c (offset) approaches. T_c (offset) rose from 82 K in the undoped sample to 106 K in the Pb-doped sample (Pb = 0.20), as can be seen. The critical transition temperatures T_c (offset) are 86 and 93 K for Pb = 0.05 and 0.1, respectively. Table (1) reveals that when the lead concentration increases ($x=0.0, 0.05, 0.1$, and 0.20), the transition temperature T_c , the volume fraction $V_{1223\text{ph}}$, and the c/a all rise. The results may be explained by stating that the addition of lead is highly successful in creating and constructing a Hg-1212 high phase by adding layers of CaO_2 and CuO_2 to the low phase of the Hg-Ba-Ca-Cu-O superconducting systems with low T_c (1201) [26,27].

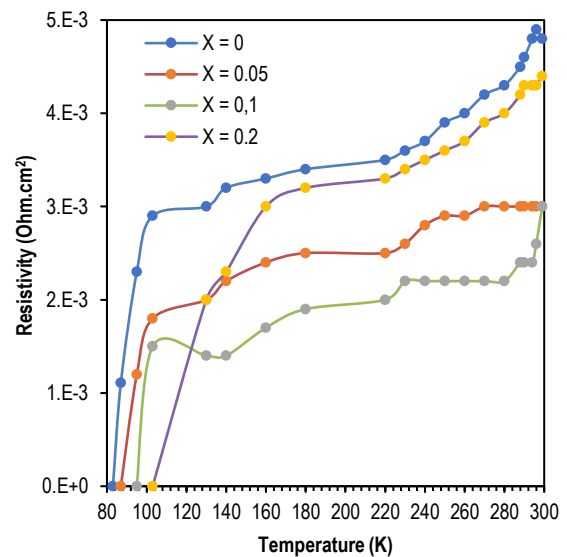


Fig. (2) Resistivity of the Hg-1212 $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ samples as function of temperature

The lead concentration and higher sintering temperatures have a significant impact on the critical temperature, potentially leading to oxygen saturation of the composition and a change in the vacancies between the (CuO) layers that affects charge transfer to the (CuO) layers [28-31]. This most likely tends to be the case of the increase in vacancies, which is reflected in the case of a random increase in the critical transition temperature, a result of which is a rise in the critical transition temperature [32], a result of which due to the absence of dispersion that takes place both at grain borders and inside individual grains [33,34].

The link between the critical temperatures and the sample's lead concentration is depicted in Fig. (3). The critical temperatures, $T_c(\text{onset})$ and $T_c(\text{offset})$, for the Hg-1212 $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ compounds increased with the lead concentration increasing, due to an increase in the percentage of 1212 phase caused by the introduction of a layer of copper oxide in phase 1201 and its transformation into phase 1212, as can be seen from this figure [35].

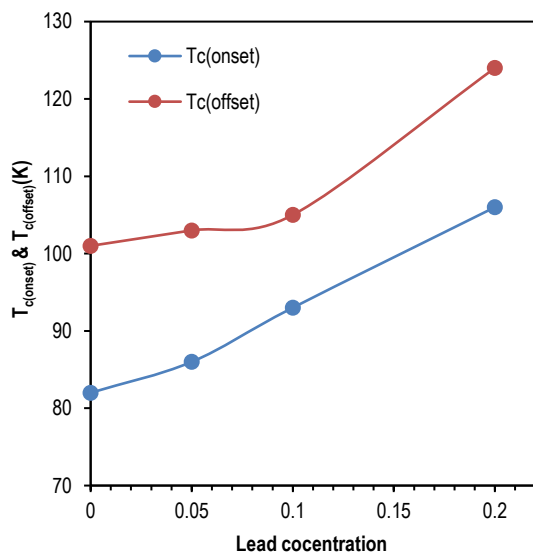


Fig. (3) The $T_c(\text{onset})$ and $T_c(\text{offset})$ of $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ samples as function of Lead concentration

Figure (4) shows the relationship between the number of vacancies and the lead concentration in the samples. It can be seen from the figure that the number of vacancies in the $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ compounds increased as the lead concentration increased. The increased holes were formed in the perovskite layers, producing increasing oxygen atoms in the (CuO) layers [36], shortening the bond length in CuO, and raising the critical scores for the superconducting (TC) materials. The structural stability brought on by the lead

insertion and the outcome of plugging the hole are the two causes of the rise in critical temperatures [37].

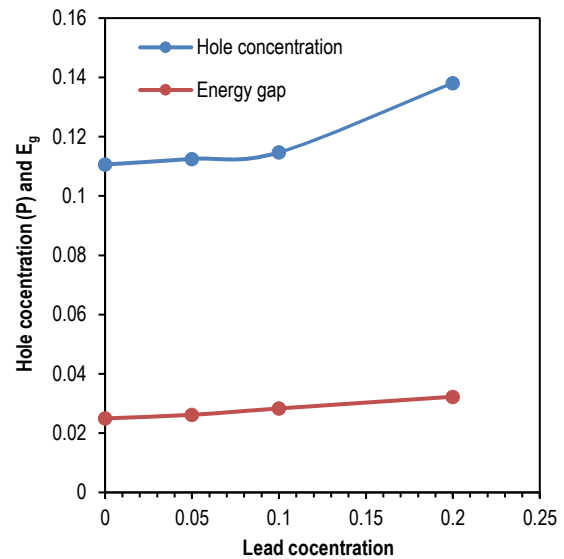


Fig. (4) Resistivity of the Hg-1212 $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ samples as function of Lead concentration

4. Conclusion

This article describes the investigation of mercury-based substituted superconductors in the series $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$ superconductor, where $x = 0, 0.05, \text{ or } 0.1$. A comprehensive study of these compounds indicated that a change in both the crystal lattice constants and the transition temperature with the increase of lead concentration in the samples. It was found that with an increase in the concentration of lead in the samples, the crystalline structure remains tetragonal structure, with an increase in the packing factor. For $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.95}\text{Pb}_{0.05}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, $\text{Hg}_{0.9}\text{Pb}_{0.1}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, and $\text{Hg}_{0.8}\text{Pb}_{0.2}\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$, the zero critical transition temperature $T_c(\text{offset})$ was found to be 82, 86, 93, and 106 K, respectively. In contrast, the onset critical transition temperature $T_c(\text{onset})$ was found to be 101, 103, 105 and 124 K respectively.

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Table (1) shows the transition temperatures T_c (offset) , T_c (Onset), lattice parameters, c/a , volume fraction V_{1212ph} and $P_{(Hole)}$ concentration for samples of $\text{Hg}_{1-x}\text{Pb}_x\text{Ba}_2\text{CaCu}_2\text{O}_{6+\delta}$. for various compositions

X	$T_{c(\text{offset})}$ (K)	$T_{c(\text{onset})}$ (K)	a(Å)	c(Å)	c/a	$V_{1212ph} \%$	E_g (ev)	$P_{(Hole)}$ concentration (cm^{-3})
0.0	82	101	3.740	12.69	3.393	77	0.024966	0.1106
0.05	86	103	3.709	12.60	3.397	81	0.026183	0.1125
0.1	93	105	3.702	12.59	3.400	83	0.028315	0.1147
0.2	106	124	3.720	12.63	3.404	88	0.032273	0.1381