



# The Effect of Humidity on Superconducting Phase of Bi-2223

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## Abstract

The ceramics specimens as superconducting phase ( $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ ) with different concentrations of Pb from (0.0-0.5) were prepared by solid-state reaction method. Superconducting samples were exposed to high humidity (RH 75% at 25°C) for seven weeks time interval. The humidity has a negative effect on the transition temperature of superconductor phase .It destroys the superconducting phase and the samples were converting to insulator.

**Keywords:** Superconductor, Bi-2223, HTC phase, Humidity effect

## Introduction

Since the discovery of the high temperature superconductor compound, several difficult have been encountered in manufacturing and storing these materials because of their chemical instability in the presence of water. The chemical instability of the superconducting phase both in high-humidity environments and in direct contact with water at temperature ranging from 25°C to 80°C has been the subject of several studies [1,2].

One great advantage of oxide compounds is their high resistivity which permits a decrease with about three orders of magnitude to humidity variations. Due to low cost and high durability, the use of the ceramic humidity sensors has greatly increased not only in electronic industry, but also in alimentary industry [3].

The high temperature superconductor compound is very brittle and suffers from a high degree of porosity .In order to overcome the poor mechanical properties, techniques such as degradation of these materials upon exposure to the humid environment have been applied [4].

Zheng et al. [5] studied the effect of water vapor on the microstructure and superconducting properties of BiPbSrCaCuO. They found that the crystal growth, especially around grain boundaries, is greatly enhanced by water vapor processing. As a result, the transport critical current density  $J_c$  is considerably enhanced.

Smrčková et al. [6] found that  $YBa_2Cu_3O_{7-y}$  superconductor is destroyed in few minutes by boiling water. At lower temperatures the hydrolysis reduces the volume of the superconductive phase,. Reaction of  $Bi_{1.4}Pb_{0.6}Sr_2Ca_2Cu_{3.6}O_x$  with water causes a decrease of the relative volume of both superconducting phases at a rate substantially higher for the high temperature one.

The objective of this work is to investigate the water-vapor effect on the microstructure of Bi-Pb-Sr-Ca-Cu-O superconductor upon exposure to humidity level (RH 75% at 25°C) for seven weeks time interval. After storage under this condition the specimens were characterized by X-ray diffraction (XRD), optical microscopy and electrical measurements.

## Experimental Part

Samples of  $Bi_2Pb_xSr_2Ca_2Cu_3O_{10+\delta}$  for  $x=0.0, 0.1, 0.2, 0.3, 0.4$  and  $0.5$  were prepared by solid –state reactions. Mixing appropriate amounts of  $Bi_2O_3, PbO, Cu_2O, CaCO_3$  and  $Sr(NO_3)_2$  develop  $Bi_2Pb_xSr_2Ca_2Cu_3O_{10+\delta}$  precursor. The mixture homogenization takes place by adding a sufficient quantity of 2-propanol to form paste, during the process of grinding for about (50-60) min. The mixture was put it in an alumina crucible, calcined in a tube furnace in air that has programmable controller type (Eurptherm818) for 24 hours at 800 °C with a rate of 2°C / min. This mixture was then pressed into pellets 1.3 cm in diameter and (0.2 – 0.3) cm thick, using hydraulic type (SPECAC) under pressure of 0.5GPa. The pellets placing in alumina crucible then heated at a rate of 2 °C/min., at 850 °C. This temperature was kept constant for about 140 hours .After that, the furnace was cooled to room temperature by the same rate

Four probe dc methods at a temperature range of (80-250) K were used to measure the resistivity ( $\rho$ ) and to determine the critical temperature ( $T_c$ ). The structure of the prepared samples was obtained by using x-ray diffractometer (XRD) (Philips).A computer program was established to calculate the lattice parameters a, b, and c. The program is based on Cohen's least square method [7]. The densities ( $d_m$ ) of unit cell of the samples were calculated using the following equation [8]:

$$d_m = \frac{W_m}{N_A V}$$

Where  $W_m$  is the molecular weight,  $N_A$  is Avogadro's number and  $V$  is the volume of the unit cell.

The samples were exposed to humidity level (RH 75% at 25°C) for seven weeks time interval. After storage under this condition the specimens were characterized by X-ray diffraction (XRD), optical microscopy and electrical measurements.

## Results and Discussion

The quantity test of the element for  $\text{Bi}_{1.66}\text{Pb}_{0.2}\text{Sr}_{1.9}\text{Ca}_{1.9}\text{Cu}_{2.13}\text{O}_{10+\delta}$  and  $\text{Bi}_{1.66}\text{Pb}_{0.5}\text{Sr}_{1.9}\text{Ca}_{1.9}\text{Cu}_{2.1}\text{O}_{10+\delta}$  system were carried out by x-ray fluorescent (XRF) before and after soaking in water for seven weeks. The elemental distribution in the samples were shown in Fig.(1). This figure indicated that Pb ions have partially replaced the Bi ions in the system.

The XRD data collected from various samples before exposed to water show that all the samples are polycrystalline and correspond to high phase (2223) as a dominate phase with low phase (2212) and impurity phases which is due to the creation of the stacking faults which leads to deform the structure. The peaks intensity of the samples stored at a constant relative humidity of 75% were found to decrease and there is a shift of  $2\theta$  values if compared before exposed to water as shown in Fig.(2).

The parameters a,b and c, c/a, c/b and density ( $d_m$ ) were also calculated from the XRD analysis as shown in Table (1). This table shows a variation of a,b and c parameter for  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  system, in comparison with the Pb free system. Indeed the deformation in the lattice parameters, as a result of substitution or deficiency of some atoms, adjusts the amount of charge transfer from Bi layer to Cu layer; this will be a driving force to the pairing generation of superconductor holes forming bosons which are the current carriers of the superconductor [9]. A deterioration of the structure was observed for samples after exposed to water, specially samples with  $x=0.5$  as shown in Fig.2d.

Also it is found from the Table an enhancement of the density value, which attribute to the decreases of the unit cell volume.

DC-four-probe resistivity measurements were performed on each ceramic specimen in the temperature range from room temperature down to boiling point of liquid nitrogen. The behavior of the samples was superconductor, there was a decreases of the resistivity with the decreasing of temperature, although in some cases a complete zero resistance could not be observed as shown in Fig.(3).

It is interesting to note that addition of Pb to the BiSrCaCuO system will improve the transition temperature from less than boiling point of liquid nitrogen for Pb free samples to 118K, for samples with  $x=0.3$ . More addition of Pb to 0.4 and 0.5 will reduce  $T_c$  to 105 and 104 respectively as shown in Table1. A certain amount of Pb is necessary for the occurrence HTP, while excessive Pb addition promotes another reaction to produce an impurity phases[10,11] which is likely to assist the formation of the low –  $T_c$  phase instead of the HTP. Indeed the amount of Pb suitable for the formation of the HTP is determined by the competition between these reactions.

Another feature of sample with  $x = 0, 0.1, 0.2$  and  $0.3$  has a tail which disappeared with Pb addition increases to 0.4 and 0.5. The reason may be attributed to the fluctuation of the oxygen content and/or existence of small amounts of the secondary phase (low and impurity phases) as indicated in the XRD analyses

During seven weeks of exposure in a humid atmosphere (25°C, 75%RH), the resistive properties of the samples deteriorated with an increase of resistivity and convert to insulator. This could be explained as follows: in the beginning water adsorption, a few water vapor molecules chemisorbs on grain surfaces by a dissociative mechanism to form two surface hydroxyls per water molecule. With increasing humidity levels, water is physisorbed on top of the chemisorbed layer [12]. Fig.(4) shows an increase of weight of the sample after the first week which was exposed to humidity.

The surface topography of the samples was determined by optical microscopy in Fig.5 before and after exposed to humid atmosphere. The most reaction occurs when BiPbSrCaCuO

material comes in contact with water vapor or humidity. According to this reaction calcium reacts with water to form calcium hydroxide which subsequently reacts with the atmospheric carbon dioxide to form calcium carbonate and that destroys the superconducting phase [4].

## Conclusion

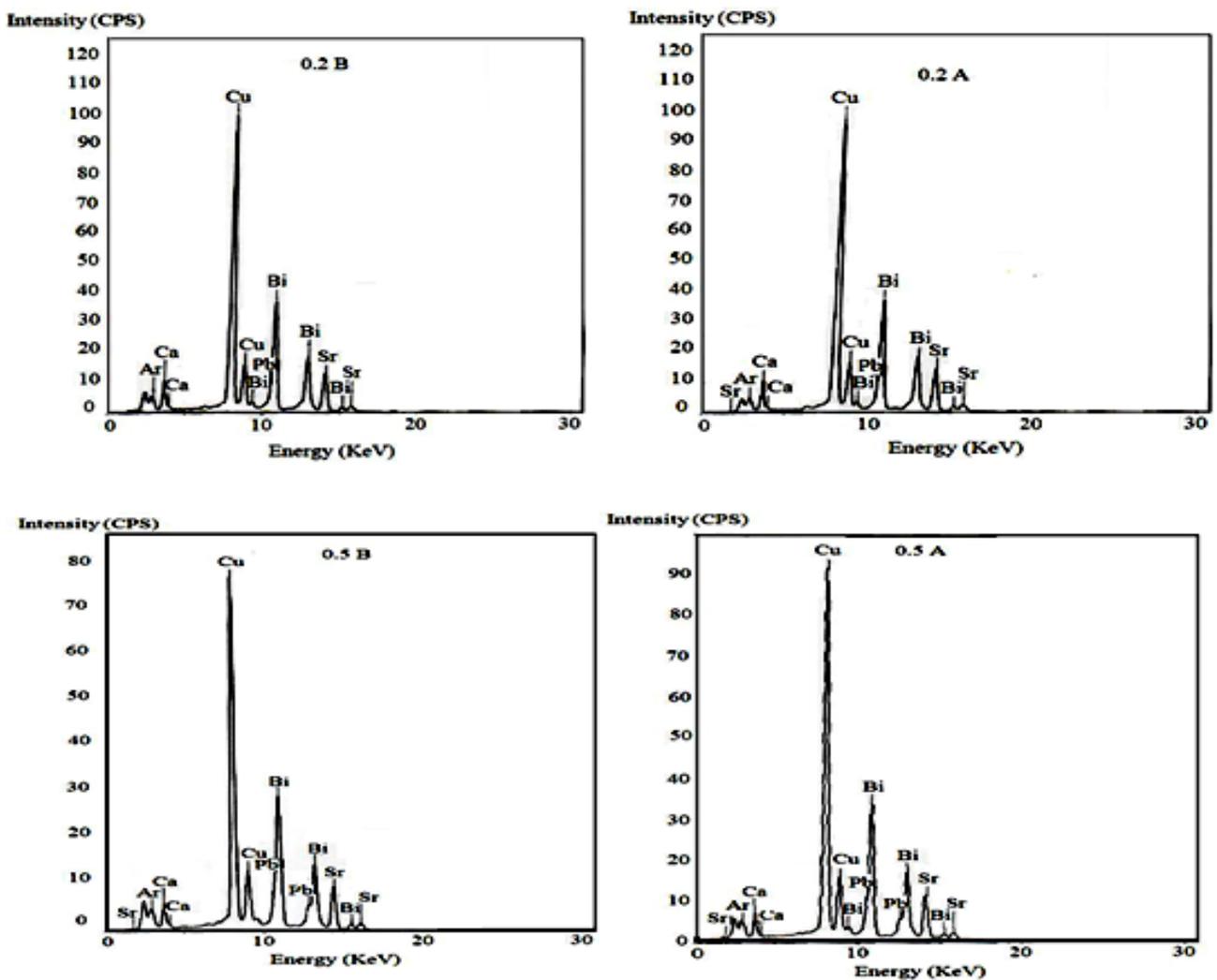
The humidity has a negative effect on the superconducting properties (structural and electrical properties). It destroys the superconducting phase and the samples were converting to insulator.

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**Table (1): Values of lattice constants, c/a, c/b, Volume, transition temperature, and density of unit cell for samples before and after exposure to water**

Pb	a (Å)	b (Å)	c (Å)	c/a	c/b	V(Å) <sup>3</sup>	T <sub>c</sub> (K)	Density(g/cm <sup>3</sup> )
0 before	5.417	5.3643	37.072	6.84364	6.91087	1077.25	<77K	1.701704
0 after	5.3875	5.3819	36.9016	6.84948	6.85661	1069.96	-----	1.713301
0.1							<77K	
0.2before	5.407	5.3627	36.9673	6.83693	6.89341	1071.91	≈100	1.70953
0.2 after	5.3367	5.3994	36.8696	6.90869	6.82846	1062.4	-----	1.790249
0.3							118	
0.4before	5.40901	5.4497	37.1187	6.86239	6.81115	1094.17	105	1.80114
0.4 after	5.3632	5.3567	37.2975	6.95434	6.96278	1071.52	-----	1.839205
0.5before	5.38146	5.39587	36.9574	6.86755	6.8492	1073.16	≈104	1.86846
0.5after	5.1461	9.08926	35.8585	6.96809	3.94515	1677.25	-----	1.195496



**Fig. (1): X-ray fluorescent for the samples  $\text{Bi}_{1.66}\text{Pb}_{0.2}\text{Sr}_{1.9}\text{Ca}_{1.9}\text{Cu}_{2.13}\text{O}_{10+\delta}$  and  $\text{Bi}_{1.66}\text{Pb}_{0.5}\text{Sr}_{1.9}\text{Ca}_{1.9}\text{Cu}_{2.1}\text{O}_{10+\delta}$  before and after exposure to water**

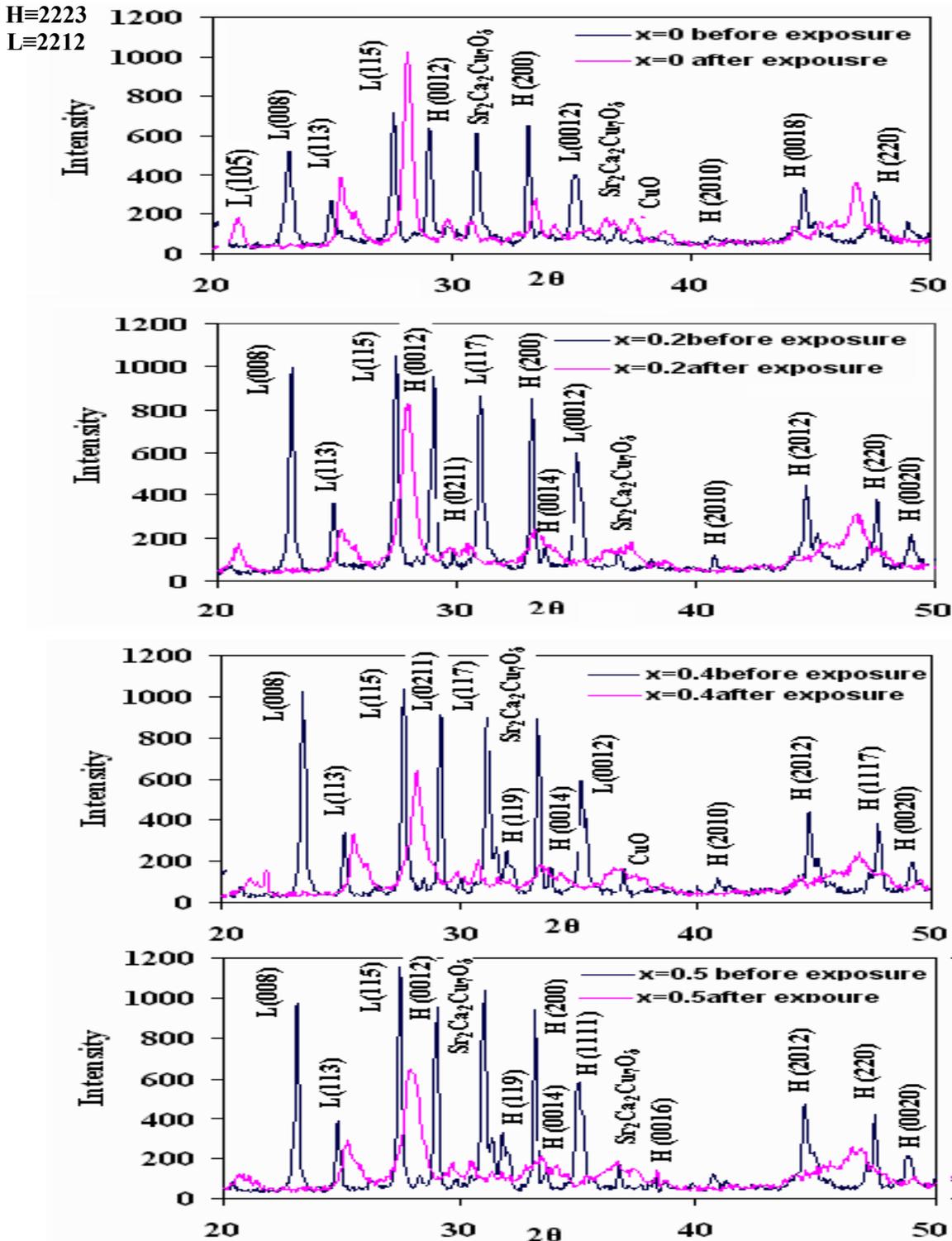


Fig.(2) : X-ray diffraction patterns for the samples  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  system before and after exposure to water

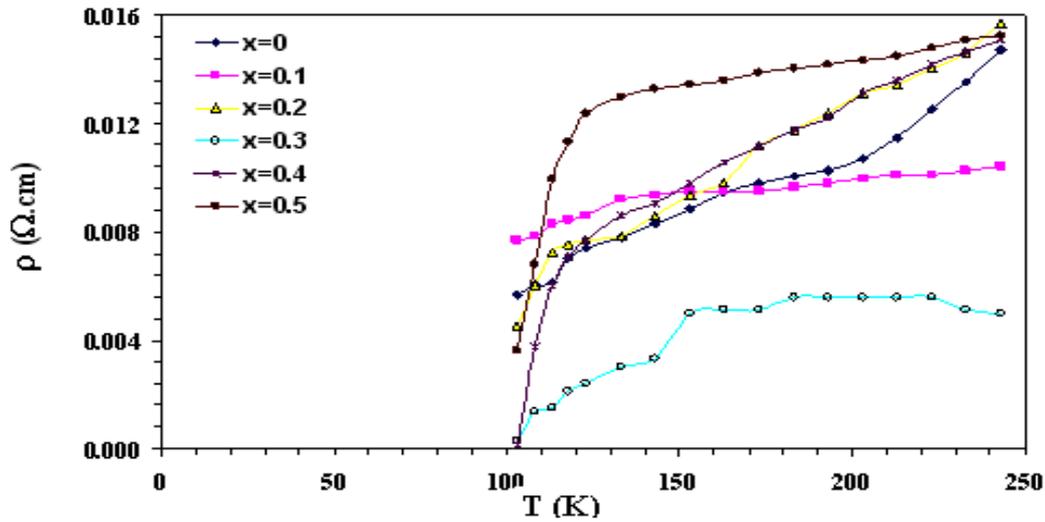


Fig.(3): Temperature dependence of resistivity for  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  system

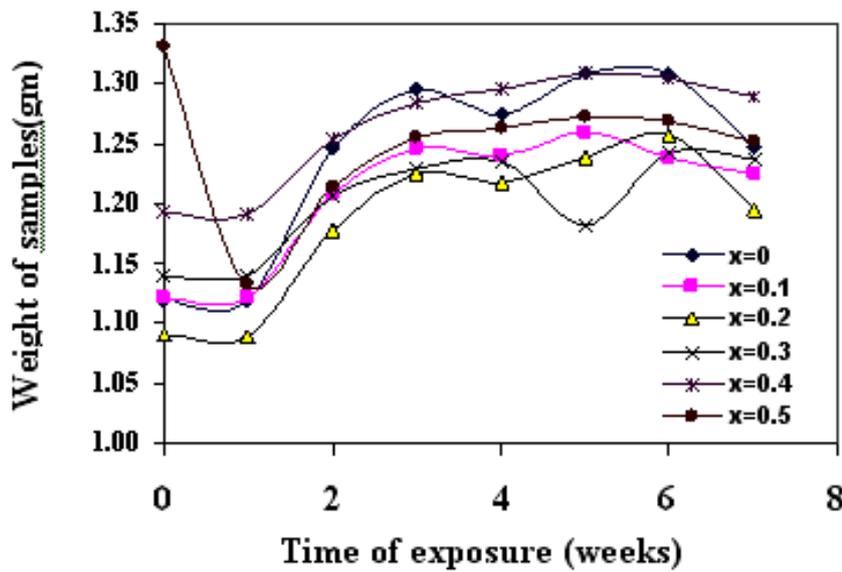
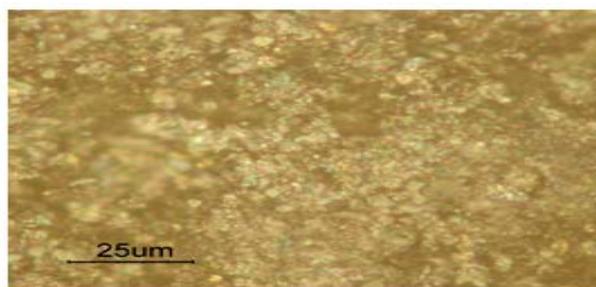
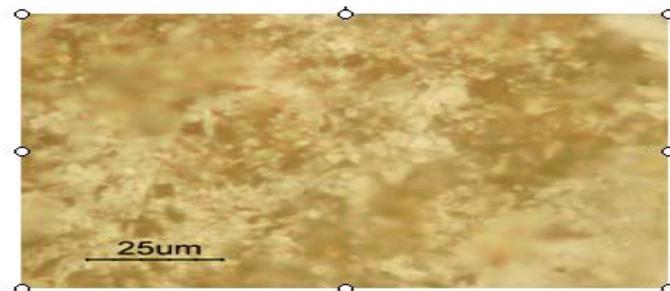


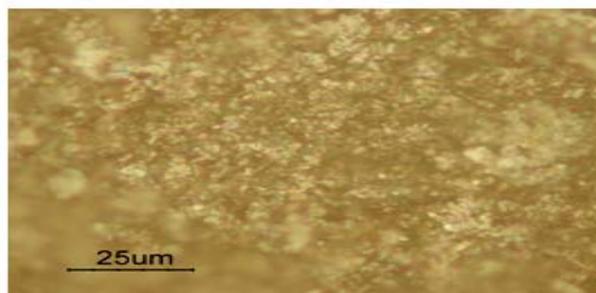
Fig. (4): Weight of the sample as a function of time of exposed to humidity for  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  system



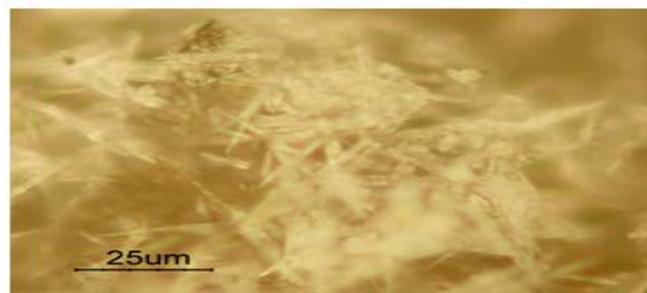
x=0 before exposure



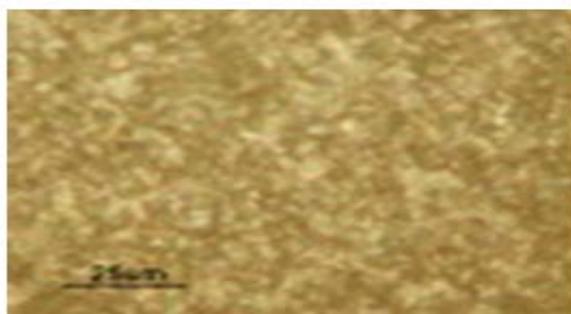
x=0 after exposure



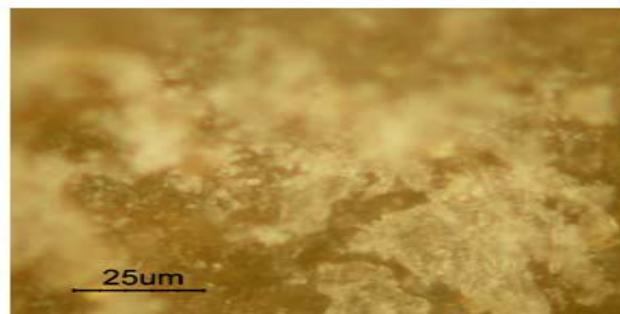
x=0.1 before exposure



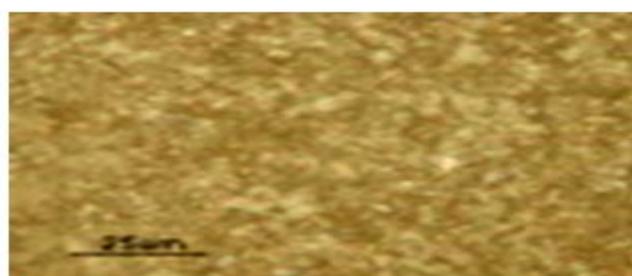
x=0.1 after exposure



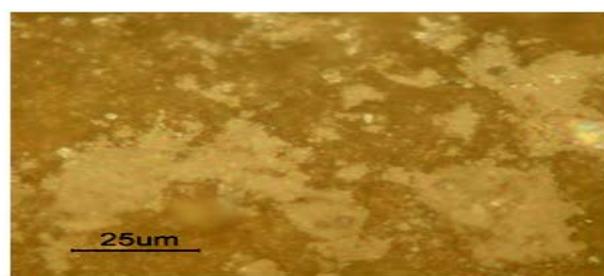
x=0.2 before exposure



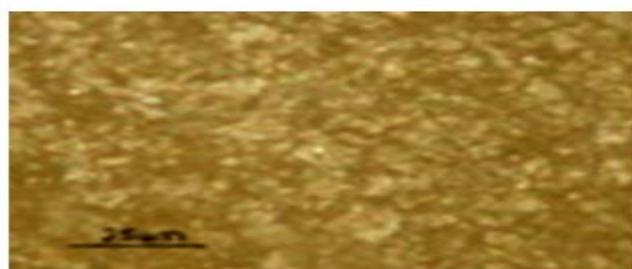
x=0.2 after exposure



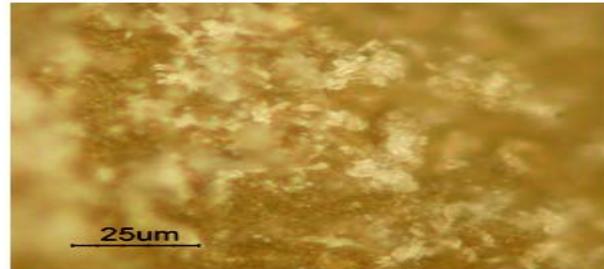
x=0.4 before exposure



x=0.4 after exposure



x=0.5 before exposure



x=0.5 after exposure

**Fig.(5): Surface topography of  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  system before and after exposure to water**

## تأثير الرطوبة في الطور الفائق التوصيل لنظام البزموت-2223

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استلم البحث في: 19 أيلول 2011 ، قبل البحث في: 2 تموز 2012

### الخلاصة

حضرت العينات الفائقة التوصيل للنظام  $\text{Bi}_2\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  ولتركيز (0.0-0.5) للخصائص باستخدام طريقة تفاعل الحالة الصلبة. تم تعريض العينات الفائقة التوصيل للرطوبة وبنسبة 75% وفي درجة حرارة 25 درجة سيليزية ومدة سبعة اسابيع. لقد كان للرطوبة تأثير سلبي على درجة الحرارة الانتقالية للطور الفائق التوصيل إذ تحطم الطور الفائق وتحولت العينات الى عازل.

**الكلمات المفتاحية:** التوصيل الفائق ، بزموت- 2223 ، الطور ذو الدرجة الحرارية العالية ، تأثير الرطوبة