

SUMMARY AND CONCLUSION

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This work had been done in an attempt to prepare superconductor materials and study its physico chemical properties. To achieve this goal, the Bi-Sr-Ca-Cu-O (BSCCO) system has been prepared by solid-state reaction using Bi_2O_3 , SrCO_3 , CaCO_3 and CuO with different composition and different annealing time. PbO and BaCO_3 were used as doped materials to study their effects on the superconducting properties. Different techniques have been utilized to study the properties of the prepared materials XRD, TG, surface morphology, density measurements, moisture effect, dc electrical conductivity measurements and dc magnetic measurements. Also the La_2CuO_4 system has been studied using the electrochemical technique.

This thesis is presented in five main chapters: introduction, theories and method of calculation, experimental, characterization of samples, results and discussion.

The first chapter is the introduction, which includes some aspects of the origin of the superconductivity. A literature survey was carried out to cover, almost, the last decade related to the proposed subject. Also it includes the aim of work.

The second chapter includes highlight on the theories and the calculation methods used in this work.

The third chapter shows the preparation of the Bi-Sr-Ca-Cu-O system with different composition and different annealing time and the preparation of La_2CuO_4 compound. This chapter also includes the different techniques used in the investigation of the samples (XRD-TG - surface morphology - density measurements - moisture effect) and measuring the electrical and magnetic properties. The preparation of La_2CuO_4 ring-disk electrodes has been reported.

The fourth chapter contains the samples characterization using the prior mentioned techniques (XRD, TG, surface morphology, density measurements and moisture effect).

The fifth chapter divided into two parts:

Part (A) includes the temperature dependence of the electrical resistance for all the prepared samples. Also this part contains the results of magnetic measurements. The results were discussed and correlated with composition of the samples, and the effect of the annealing time. Comparing the obtained results from dc electrical measurements and magnetic measurements with the XRD analysis to

identify the superconducting properties. The effect of doping on the samples using Pb and Ba has been studied.

Part (B) includes the studying of the dissolution behavior of La_2CuO_4 by using electrochemical techniques in aqueous media under open circuit conditions at 298 K. Rotating ring-disk electrode (RRDE) techniques were employed in collection (product detection) and shielding (reactant removal) measure modes to monitor the surface chemistry, including a new approach not involving separate disk electrode H^+ reduction measurements as required before. Also converted the La_2CuO_4 semiconductor material to a superconductor material ($\text{La}_2\text{CuO}_{4+x}$) at room temperature using a unique electrochemical oxidation method. The electrochemical efficiency of anodic formation of the superconductor phase, $\text{La}_2\text{CuO}_{4+x}$, in alkaline medium has been studied.

The main conclusions of this work are the following:

- 1- Thermal analysis shows that all the samples are thermal stable up to 1023 K.
- 2- The XRD analysis for all the prepared samples indicates the presence of the two superconducting phases; 2212, 2223 and a semiconducting phase ; 2201.

- 3- A diffraction peak at 5.9° which corresponds to the double Cu-O layers have been observed for all the samples, 2212, this explains the superconducting behavior for the samples.
- 4- The increase in the intensity of 4.8° peak corresponded to the increase of diamagnetic susceptibility of the high T_c phase, which is clear in sample EM5, doped with Ba.
- 5- The surface morphology of the samples indicates that all the samples have the same type of surface and the addition of Pb^{2+} or Ba^{2+} did not change the type of crystal formed but causes a change in the size of crystals, it means that they can be used as nucleation centers for superconducting phases.
- 6- The dc electrical measurements showed a metallic behavior for all the samples from 300 K to 120 K.
- 7- The change in the composition or the annealing time plays an important role in the preparation and properties of the superconductor materials.
- 8- Using doping substance as Pb and Ba can be enhancement the value of T_c .
- 9- The addition of lead to 0.3 atomic ratio increases the T_c but more addition of lead induces an insulator phase which causes an

increase in the resistance at temperature above T_c . This can be observed from the resistance values at 150 K for the samples containing lead.

- 10- All samples heated at 1123 K were superconducting, with bulk of their resistance drop between 120 and 135 K, however the $T_{c,off}$ varied with thermal history.
- 11- The maximum $T_{c,off}$ was 73 K in a sample EM2 heated at 1123 K For 10 days and slowly cooled, and the minimum was 52 K in a sample EM3 heated at 1123 K for 3 days and slowly cooled.
- 12- The magnetic measurements showed a diamagnetic behavior for all the samples.
- 13- The dissolution reaction of La_2CuO_4 with protons is limited by mass transport.
- 14- New simplified method for determining the number of protons in the dissolution reaction can be made.
- 15- La_2CuO_4 has chemical reaction properties in common with those of $Ba_2YCu_3O_7$ and is much more reactive than CuO .
- 16- The rate of formation of superconductor phase is very slow, the maximum thickness of the superconductor formed was about 163 nm thick from short time experiments.