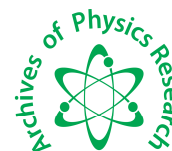




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Crystalline structural changes in Na-doped Y-Ca-Cu-O oxides

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ABSTRACT

Na-doped $YCa_2Cu_4O_{7-\delta}$ oxides are prepared by solid state diffusion of heating the reacting oxides (standard ceramic technique). The X-ray diffraction measurements show that the Na-doped $YCa_2Cu_4O_{7-\delta}$ is single phase orthorhombic structure by annealing below 900°C under oxygen atmosphere. It is observed that, increase in Na content, the orthorhombic distortion decreases.

Keywords: Oxide superconductor, orthorhombic structure, tetragonal symmetry.

INTRODUCTION

Ever since the discovery of high temperature superconductivity in the Y-Ba-Cu-O system [1] several isovalent and aliovalent substitutions for Y and Ba were made by different groups in order to investigate the structure–property relationships in these compound. Investigation on co-substitution of Ca^{2+} for Y^{3+} and La^{3+} for Ba^{2+} in Y-Ba-Cu-O system, a new tetragonal $CaBaLaCu_3O_{7-\delta}$ was discovered independently by two groups [2, 3]. After extensive survey on cuprate oxide superconductors, we thought that the field is interesting with replacement of ‘Ba’ by ‘Ca’ and partial substitution of ‘Na’ for ‘Y’ in the oxide having general chemical composition $Y_{1-x}Na_xCa_2Cu_3O_{7-\delta}$ for $x = 0.0, 0.1, 0.2, 0.3$ & 0.4 . The purpose of present work is to study the effect of change of concentration of ‘Na’ for ‘Y’ in $Y_{1-x}Na_xCa_2Cu_3O_{7-\delta}$ and to characterize these for structural point of view. The oxides with general chemical formula $Y_{1-x}Na_xCa_2Cu_3O_{7-\delta}$ for $x = 0.0, 0.1, 0.2, 0.3,$ & 0.4 are prepared by oxide method. Each sample is prepared with same atmosphere and the identical preparational procedure is adopted for the all samples studied here. Phase purity is checked by X-ray diffraction analysis. X-ray diffractometry is used for identification of these samples using same X-radiation.

MATERIALS AND METHODS

The simplest method of preparing oxide superconductor in a sintered polycrystalline state is a solid state diffusion of heating the reacting oxides (standard ceramic technique), which are easily decomposed to oxides. In this method, the impurity addition is avoided. The formation of compounds depends on many factors such as particle, size of reactants, sintering temperature and

atmosphere etc. This method for preparation of oxide compounds is extensively used due to its simplicity [4].

All the compounds having the chemical composition $Y_{1-x}M_xCa_2Cu_3O_{7.8}$ (where $M = Na$ for $x = 0.0, 0.1, 0.2, 0.3,$ and 0.4) are prepared by using high purity annular grade oxide under identical condition. The reacting oxides $Y_2O_3, Na_2O_3, CaCO_3$ and CuO (All 99.9 % pure) first heated individually in air at $200^\circ C$ for 5 - 6 hours to remove the moisture. They are intimately mixed together in the proper molar ratio. To achieve homogeneity and for uniform grain size in polycrystalline form, the mixtures are continuously ground about 5 - 6 hours under A. R. grade acetone in a agate mortar. After grinding the mixture, they are dried in air and heated in air at $800^\circ C$ for 24 hours in an electrical furnace. The resulting specimen following the reaction were ground and retreated at $900^\circ C$ for 24 hours to obtain a homogenous single phase sample. The resultant black product was reground and cold pressed into pellets, which are sintered in air at $900^\circ C$ and $950^\circ C$ for 24 hours. These pellets were annealed under oxygen flow at $450^\circ C$ for 12 hours followed by slow cooling at the rate of $1^\circ C/min$ until room temperature is reached.

For the pelletization, the resulting mixture obtained was added with 5 % polyvinyl acetate (PVA) solution in AR grade acetone as a binder and mixed thoroughly. This PVA mixed sample is then pressed in hydraulic press for about 5 minutes. The pellets thus obtained are heated in an electric furnace at about $300^\circ C$ for about 4 hours to remove the binder and then PVA removed pellets are fired at higher desired temperature to obtain the resulting oxides. These pellets thus obtained are crack free and hard. One pellet of each sample was then finely powered in an agate mortar for characterization. Philips W-1710 X-ray diffractometer and using Cu-K radiation in the 2θ range of 0-60 is carried the XRD.

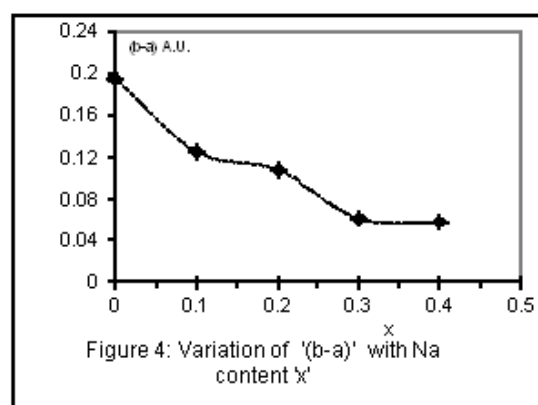
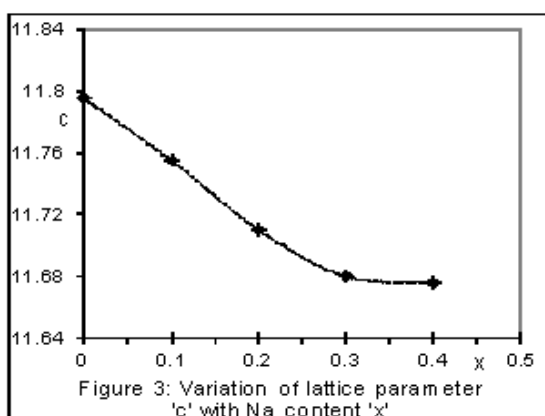
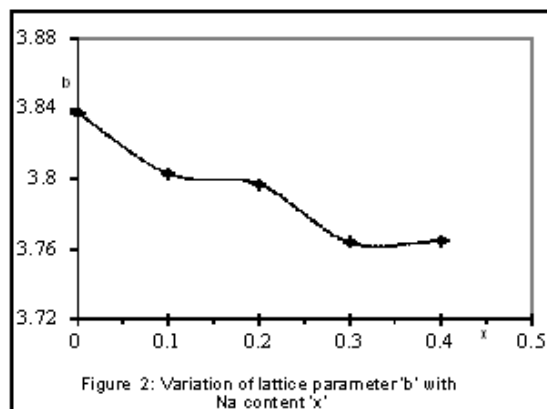
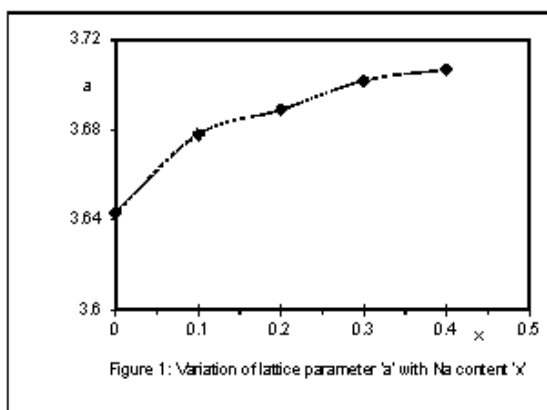
RESULTS AND DISCUSSION

The XRD pattern are taken on sintered $Y_{1-x}Na_xCa_2Cu_3O_{7.8}$ sample for $x = 0.0, 0.1, 0.2, 0.3$ and 0.40 . As all the oxides have same chemical formula with changes in cations which are not differing heavily in ionic sizes, all of them are expected to have a similar crystal structure. With this idea in mind, all the peaks in the XRD pattern of samples have been indexed for orthorhombic structure. The observed 'd' values found to be in good agreement with the calculated values. The lattice parameters, unit cell volume and orthorhombic distortion which is defined as $(b-a)$ corresponding to the concentration of rare earth 'Na' substituted for Y is shown in Table 1.

Table 1: Structural parameters

Composition (x)	Unit cell parameters			Unit cell Volume (\AA^3)	Orthorhombic distortion (b-a) (\AA)
	a (\AA)	b (\AA)	c (\AA)		
0.0	3.643	3.838	11.795	164.915	0.195
0.1	3.678	3.803	11.755	164.442	0.125
0.2	3.689	3.797	11.71	164.023	0.108
0.3	3.702	3.764	11.680	162.75	0.062
0.4	3.707	3.765	11.676	162.96	0.058

The variation of lattice parameters 'a', 'b' and 'c' with sodium content 'x' is shown in Figure 1, Figure 2 and Figure 3 respectively.



The lattice parameter 'a' increases with the Na content 'x' while 'b' decreases with increase in content 'x' up to $x = 0.3$ but increases for $x = 0.4$. Further, the lattice parameter 'c' decrease with the Na content $x \leq 0.4$. This is due to the geometrical considerations. All the compounds maintain a orthorhombic perovskite Structure. 'c' parameter and unit cell volume decrease drastically with increasing sodium concentration x up to 0.2. It may be due to introduction of Na^+ which has the comparable ionic radius as that of Y^{3+} . But remains nearly constant for system with $x \geq 0.3$. On the other hand, electrons will be introduced into antibonding Cu-O orbitals on the a-b plane by the substitution which tends to expand the a-dimension [5]. This was attributed to comparable size of Na^+ and Y^{3+} which has pronounced effect on 'c' parameter because the lattice parameter 'c' is approximately three times 'a' and 'b'. It appears that among the system studied, it attains maximum contraction at sodium content $x \approx 0.3$. A similar observations was made by Ono et al [6] where 'c' and 'a' decreases drastically for the concentration of $\text{Sr}^{2+} \leq 0.5$ on Ba side and nearly constant for system with $x > 0.5$. Similar results have been reported for $\text{La}_{4-x}\text{Ca}_x\text{Ba}_3\text{Cu}_7\text{O}_2$ [7], $\text{La}_3\text{Ba}_{4-x}\text{Ca}_x\text{Cu}_7\text{O}_2$ [8], $\text{La}_{3-x}\text{Ca}_{2x}\text{Ba}_{3x}\text{Cu}_6\text{O}_2$ [9], $\text{YBa}_{2-x}\text{K}_x\text{Cu}_3\text{O}_{7-\delta}$ [10] and $(\text{Y}_{1-x}\text{Sr}_x)(\text{Ba}_{1.5}\text{Sr}_{0.5})\text{Cu}_3\text{O}_{7-\delta}$ [11].

The variation orthorhombic distortion (b-a) with Na content x is shown in Figure IV. All the oxides crystallize in orthorhombic symmetry but the extent of orthorhombic distortion decrease with x . It is seen that the lattice parameter 'a' increases with increase in 'x' and may approach 'b' at higher values of 'x' and may result the phase transition from orthorhombic to tetragonal symmetry.

The similar is reported in Raman et al [10] for $\text{YBa}_{2-x}\text{K}_x\text{Cu}_3\text{O}_{7-\delta}$. The decrease in (b-a) is may be due to decrease in oxygen content as a consequence of increase in Na concentration.

CONCLUSION

The $\text{YCa}_2\text{Cu}_4\text{O}_{7-\delta}$ compounds can be prepared at an ordinary pressure of oxygen. The compounds crystallize to orthorhombic system with single phase. The lattice parameter 'c' is roughly three times the parameter 'a' and 'b'.

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