TECHNICAL ARTICLE

Synthesis, structural and electrical characterizations of $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$

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A new compound $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ($ErSr_5Ni_{2.4}Cu_{0.6}O_{11}$) was prepared using the conventional solid state method and annealed at 1423 K in 1 atm of oxygen gas flow. The oxygen non-stoichiometry ($\delta = 0.47$) was determined by iodometric titration. Rietveld refinement using powder X-ray diffraction data confirms that the sample adopts the K_2NiF_4-type structure (space group $I4/mmm$ ($Z = 2$), $a = 3.760\,56(4)$ and $c = 12.3889(1)$ Å). The final reliability factors were: $R_{wp} = 10.75\%$, $\chi^2 = 2.51$, $R_p = 14.80\%$, $R_B = 4.77\%$ and $R_F = 2.73\%$. Four probe electrical resistivity measurements were performed vs. temperature in the range of 320–540 K. A semiconducting behaviour over the whole range of temperature, with a maximum conductivity of 0.026 S cm^{-1} is observed at 439 K. © *2012 International Centre for Diffraction Data* [doi:10.1017/S0885715612000644]

Key words: Rietveld refinement, K_2NiF_4-type structure, electrical resistivity, $ErSr_5Ni_{2.4}Cu_{0.6}O_{11}$, iodometric titration

I. INTRODUCTION

Oxides of the A_2BO_4 type with A = lanthanides, B = 3d transition metals possessing K_2NiF_4-type structure have been extensively studied in recent years because of their catalytic activities and electrical and magnetic properties. A unique set of these properties makes them useful as electrode materials in different electrochemical devices. Further investigations on these oxides have been conducted to find appropriate compositions with the best set of necessary properties. In fact, $La_{1.85}Ba_{0.15}CuO_4$, the barium-doped analogue of La_2CuO_4 was the first high-T_c oxide discovered with T_c at 30 K (Bendorz and Müller, 1986).

Many authors have reported studies on nickel oxides having the same structure. In particular, extensive investigations have been conducted on the structural, magnetic and electrical properties of $La_{2-x}Sr_xNiO_{4-\delta}$ (Gopalakrishnan *et al.*, 1977; Sreedhar and Rao, 1990; Takeda *et al.*, 1990) and $Nd_{2-x}Sr_xNiO_{4-\delta}$ (Brian *et al.*, 1990; Takeda *et al.*, 1992) solid solutions. We are more interested in the mixed oxides based on neodymium rare earth, and have previously studied the series $NdSrNi_{1-x}Cu_xO_{4-\delta}$, $0 \leq x \leq 1$ (Chaker *et al.*, 2004, 2006). A systematic substitution of Ni by Cr in the $NdSrNiO_4$ parent compound was reported in our previous works, and we have synthesized new compounds in the $NdSrNi_{1-x}Cr_xO_{4-\delta}$, with $0 \leq x \leq 1$ by the solid state method (Chaker *et al.*, 2010) and by the modified sol gel method and subsequent annealing in 1 atm of Argon gas flow (Jammali *et al.*, 2010). Therefore, we realized that experiments in the fully oxygenated atmosphere to prepare an oxygen-deficient compound ($Ln_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$), always produce multiphase samples, but the preparation under 1 atm of argon gas flow resulted in the formation of the pure compound.

A study of the $Ln_{2-x}Sr_xNiO_{4-\delta}$ system with smaller Y^{3+} cation leads to the phase $YSr_5Ni_3O_{11}$ (James and Attfield, 1993). In one of our previous investigations (Chaker *et al.*, 2007) of the quaternary system Ln_2O_3–SrO–NiO–CuO, we have determined similar phases of $LnSr_5Ni_{2.4}Cu_{0.6}O_{11}$ that form with the small lanthanides Ho, Er, and Dy. We have described the electrical properties and crystal structures of $Dy_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ (Hamdi *et al.*, 2011b) and $HoSr_5Ni_{2.4}Cu_{0.6}O_{11}$ (Chaker *et al.*, 2007), all of them experiencing a high-temperature semiconductor. To the best of our knowledge, there are no reports on the preparation, crystal structure or physical properties of $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$. In the present work, we describe the synthesis of the new compound $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_4$, along with the structural characterization and the resistivity measurements at high temperature.

II. EXPERIMENTAL

A. Synthesis

The $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ($ErSr_5Ni_{2.4}Cu_{0.6}O_{11}$) sample was prepared in a polycrystalline form using the conventional solid state method. Before use, Er_2O_3 (Aldrich 99.99%) was calcined at 1373 K for 9 h in air to remove any carbonate and bicarbonate impurities. NiO and CuO (Aldrich 99.99%) were used as obtained. Stoichiometric amounts of Er_2O_3, $SrCO_3$, NiO and CuO were thoroughly ground together and calcined at 1223 K for 36 h. The obtained powder sample was pelleted, then sintered in a tube furnace at 1423 K under flowing oxygen to maximize the oxygen content for 48 h and then cooled slowly to room temperature. The sample is repelletized and reheated several times for the same period until no further changes in the X-ray diffraction pattern were observed.

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B. Data collection and electrical measurements

To identify the phase composition and to refine the unit cell parameters, the sample was examined by X-ray diffraction using BRUKER AXS D8 ADVANCE diffractometer with a monochromatic $CuK\alpha_1$ radiation ($\lambda = 1.540\,56$ Å) obtained with an incident-beam curved germanium monochromator. Data were collected at each 0.0078° step width for 1.7 s over a 2θ range from 8.0000° to 120.0063°. Pattern matching and Rietveld refinement were performed using FullProof software (Rodriguez-Carvajal, 1990).

Electrical direct current resistivity measurements were performed on sintered pellets using a Lucas Labs 302 four point probe with a Keithley 2400 digital Source Meter (Keithley Instruments Inc., Cleveland, Ohio). Measurements were performed in the temperature range of 320–540 K.

The oxygen content of the compound was indirectly determined at room temperature after calculation of the valence average of the transition metal ions obtained by iodometric titration under flowing nitrogen gas taking into account that Ni^{3+} and Cu^{3+} can be present in the sample with Ni^{2+} and Cu^{2+}. In fact, 60 mg sample was dissolved in a solution of 6 M HCl in the presence of an excess KI, leading to reduction of Ni^{3+} to Ni^{2+} and all the copper ions (Cu^{3+} and Cu^{2+}), to Cu^+ ions with the formation of iodine that was titrated with a $Na_2S_2O_3$ solution using starch as an indicator. The sodium thiosulfate solution was standardized using pure copper wire.

III. RESULTS AND DISCUSSION

A. Crystal structural and oxygen stoichiometry

The exact temperature, the gas atmosphere, and the reaction time are considered essential to obtain a pure phase containing no traces of the starting materials. In fact the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ sample was successfully synthesized under flowing oxygen at 1423 K. Inspection of the X-ray patterns at intermediate stages of the synthesis appeared to indicate that an impurity phase identified as Er_2SrO_4 (PDF-00-046-60133) (ICDD, 1994) still persists even after a multitude of sintering. Therefore, a biphasic Rietveld refinement was applied for the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ sample. Quantitative X-ray analysis of the sample by FULLPROOF program, using Brindley's procedure (Brindley, 1949) indicates a proportion less than 1% of Er_2SrO_4. Hence, this impurity is considered to have a negligible effect on the stoichiometry of the material. The refinement showed a good fit between the observed and the calculated patterns and the cell dimensions obtained from this refinement are given in Table I.

Profile analysis of X-ray diffraction pattern for $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ compound was carried out on the

basis of a tetragonal system (space group $I4/mmm$) by analogy with similar phases formed with Ho (Salwa et al., 2011) and Dy (Chaker *et al.*, 2007). The background was fitted with a linear interpolation between 100 chosen points. The lattice parameters deduced from the X-ray powder diffraction profile refinement are found to be: $a = 3.760\,56(4)$ and $c = 12.3889(1)$ Å.

The stability of the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ compound having a K_2NiF_4-type structure can be discussed in terms of the tolerance factor value defined by Goldschmidt (Ganguly and Rao, 1984), as:

$$ t = \frac{\dfrac{0.33 r_{Er^{3+}} + 1.67 r_{Sr^{2+}}}{2} + r_{O^{2-}}}{\sqrt{2}\left[0.8 r_{Ni^{3+}} 0.2 r_{Cu^{2+}} r_{O^{2-}}\right]}. $$

The K_2NiF_4-type structure is stable over the range $0.866 \le t < 1$. The T (tetragonal) structure exists for $0.88 \le t \le 0.99$ and the T/O (tetragonal/orthorhombic) structure is present for $0.866 \le t < 0.88$. Based on Shannon's ionic radii (Shannon, 1976), ($r_{Er}^{3+} = 1.062$ Å, $r_{Sr}^{2+} = 1.31$ Å in a nine-fold coordination and $r_{Ni}^{3+} = 0.56$ Å, in a low spin case, $r_{Cu}^{2+} = 0.73$ Å and $r_O^{2-} = 1.4$ Å in a six-fold coordination), the calculated tolerance factor for $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ is $t = 0.9465$, this value is included in the tetragonal symmetry stability range.

Consequently, Rietveld refinement of $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ X-ray data was performed to confirm the K_2NiF_4-type structure. The structural refinement was carried out in the space group $I4/mmm$ starting with atomic positions taken from $Ho_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ($HoSr_5Ni_{2.4}Cu_{0.6}O_{11}$) (Chaker *et al.*, 2007), with Er/Sr and O(2) atoms situated at special positions 4e with coordinates (0, 0, z). The Ni and Cu atoms are located at (0, 0, 0) in the 2a site, and the O(1) atoms at (0, ½, 0) in the 4c site. Atomic positions were refined for all atoms, together with scale factor and profile parameters. It is well known that in Rietveld refinements of heavy-atom structures, using X-ray diffraction data, much problematic intensity may be hidden in the thermal displacement factors of relatively light atoms (Hill, 1992; Hamdi *et al.*, 2011a, 2011b). That is why we undertook the refinement of oxygen atoms with fixed occupancies at 0.125 and thermal displacement factors fixed to values higher than those of heavy atoms. It was also impossible to refine the Ni/Cu ratio, owing to the similar X-ray scattering powers of these elements. Refinements of the physically sensible isotropic temperature factors were unstable, but good results were obtained by refinement of the overall isotropic displacement factor. Finally, refinement of the preferred orientation correction in the [001]-direction led to a significant decrease of χ^2, and a considerable improvement in the visual quality of the fit. A final refinement converged to $R_{wp} = 10.75\%$, $\chi^2 = 2.51$, $R_p = 14.80\%$, $R_B = 4.77\%$ and $R_F = 2.73\%$.

To determine the oxygen non-stoichiometry, we carried out an iodometric titration using the $Na_2S_2O_3$ standard solution. The oxygen non-stoichiometry (δ) is directly correlated with the Ni^{3+} and Cu^{3+} content according to the formulation $Er_{0.33}Sr_{1.67}(Ni_{1-\tau}^{3+}Ni_\tau^{2+})_{0.8}(Cu_{1-\tau}^{3+}Cu_\tau^{2+})_{0.2}O_{4-\delta}$, with $\delta = 0.33 + (1/2)\tau$. The content $(1 - \tau)$ of the average of cations transition metals Ni^{3+} and Cu^{3+} was determined by iodometric titration. I^- anions reduce Ni^{3+} to Ni^{2+} and all the copper ions (Cu^{3+} and Cu^{2+}) to Cu^+. The titration of the resulting I_2, by a solution of $Na_2S_2O_3$ sodium thiosulfate led to determining the value $\tau = 0.28$ and therefore $\delta = 0.47$ after considering the average of cations transition metals Ni^{3+} and Cu^{3+}. The

TABLE I. Unit cell for $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$.

Chemical composition	$Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ($ErSr_5Ni_{2.4}Cu_{0.6}O_{11}$)
Space group	$I4/mmm$
a (Å)	3.760 56(4) Å
c (Å)	12.3889(1) Å
V (Å^3)	175.201(2) Å^3
Z	2
D_x (g cm^{-3})	6.164

obtained results indicate that the compound is oxygen deficient.

B. Structure description

The observed, calculated and difference profiles for the Rietveld refinement of $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ compound are shown in Figure 1. An enlargement of the fit to the 2θ region between 26° and 46° 2θ is also presented in Figure 1. The structural refined parameters for $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ are summarized in Table I.

As shown in Figure 2, the structure can be described as an ordered intergrowth of alternating perovskite (Er/Sr)(Ni/Cu) O_3 and rock salt (Er/Sr)O layers stacked along the tetragonal c-axis. The $(Ni/Cu)O_6$ octahedra shares corners in the ab-plane forming a two-dimensional array of (Ni/Cu)–O–(Ni/Cu) bonds which is responsible for a variety of interesting physical phenomena, as for example, the anisotropic electrical transport and magnetic exchange interactions (Bassat *et al.*, 1987; Buttrey and Honig, 1988).

C. Electrical conductivity

To obtain dense ceramic pellets for electrical measurements, a powder sample of the material was grounded (in ethanol). A pellet (13 mm in diameter and 1.2 mm thick) was prepared by uniaxial pressing (100 MPa). It was then sintered at 1173 K for 4 h to obtain a disk with high density. The total electrical conductivity σ_e of the sintered ceramic was determined, under air using the four-probe technique, in the temperature range of 320–540 K. The corresponding curve to σ_e vs. 1000/T is reported in Figure 3. First, the conductivity increases with temperature to reach a maximum observed between 425 and 439 K then, above this temperature, the conductivity decreases, owing to the decrease of the oxygen content and correspondingly of the density of Ni^{3+} and Cu^{3+} carriers (Bassat *et al.*, 1994; Boehm *et al.*, 2005). We focus our study on the conduction mechanism only in the temperature range up to 439 K (where the maximum of conductivity is achieved). Figure 4 shows the relationship of $Ln(\sigma T)$ vs. 1000/T for the temperature range of 320–439 K. The increase of $Ln(\sigma T)$ with increasing temperature indicates that the compound
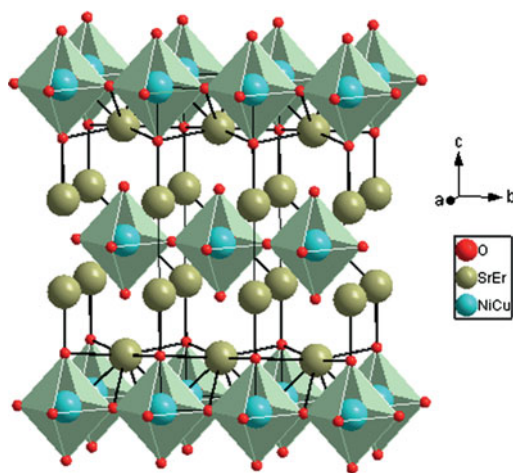

Figure 2. Crystal structure of the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ sample with the tetragonal K_2NiF_4-type structure.
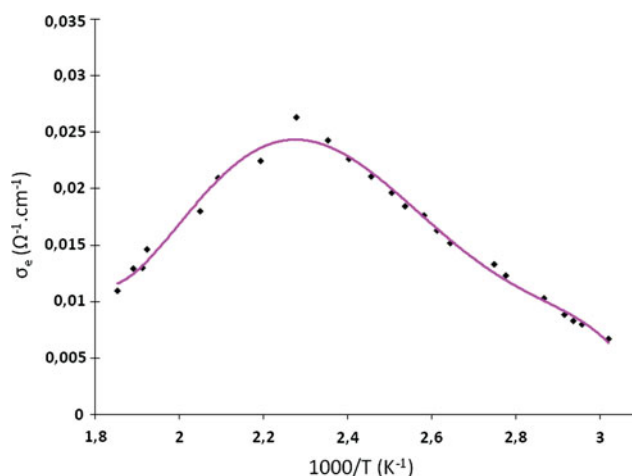

Figure 3. Thermal variation of electronic conductivity σ_e for the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ compound ($320 \leq T(K) \leq 540$).

has a p-type semiconductor behaviour over the chosen temperature range. To determine the conduction mechanism, we tried to use the thermally activated adiabatic small polaron hopping as a conduction model (Mott and Davis, 1971;
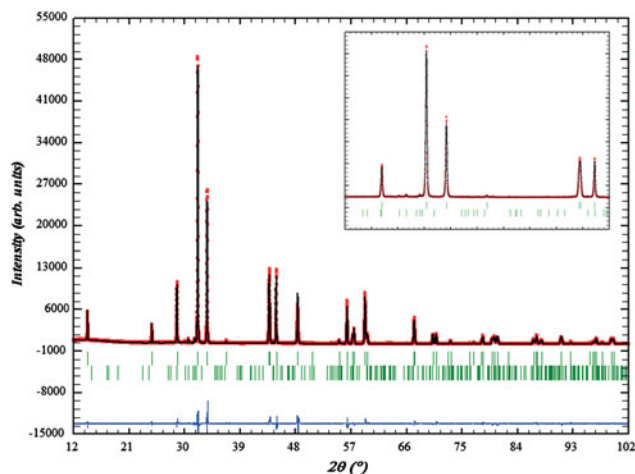

Figure 1. Observed, calculated and difference profiles for Rietveld refinement of $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ compound.
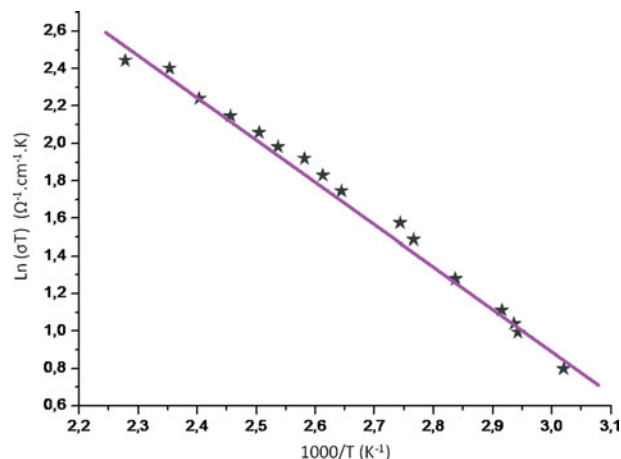

Figure 4. Arrhenius relations of $Ln(\sigma T)$ vs. 1000/T for the $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$.

Long *et al.*, 1991; Iguchi *et al.*, 1998; Tan *et al.*, 2008):

$$\sigma = \sigma_0 T^{-1} \exp\left(\frac{-E_a}{k_B T}\right),$$

where E_a is the activation energy (polaron formation and hopping energy), σ_0 is a constant related to polaron concentration and diffusion. In the analysis of the simulated curve: $\mathrm{Ln}(\sigma T) = f(1000/T)$, we found that the convergence of curve fitting was achieved in the whole range of temperature. This suggests that the charge transport in $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ceramic, in the temperature range of 320–439 K, is described by an adiabatic small polaron hopping model, where activation energy, after fitting, is $E_a = 0.21$ eV.

IV. CONCLUSION

The $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ ($ErSr_5Ni_{2.4}Cu_{0.6}O_{11}$) compound was successfully formed at 1423 K in an oxygen atmosphere. Rietveld refinement using powder X-ray diffraction data shows that the title compound crystallizes in the tetragonal K_2NiF_4-type structure in space group *I4/mmm*. Results obtained by iodometric titration indicate that the compound is oxygen deficient ($\delta = 0.47$). The transport mechanism in $Er_{0.33}Sr_{1.67}Ni_{0.8}Cu_{0.2}O_{4-\delta}$ was investigated. On heating, under air, first, the conductivity increases with temperature to reach a maximum at 439 K. Above this temperature, the oxide starts to lose oxygen; consequently holes concentration is decreased and resistivity increased. The fitting shows that the adiabatic small polaron hopping model describes the experimental data in the temperature range of 320–439 K.

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