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Synthesis and characterisation of $\text{Nd}_{1.95}\text{Sr}_{0.05}\text{FeO}_{4+\delta}$ cathode material for intermediate temperature solid oxide fuel cell

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Abstract

Intermediate temperature solid oxide fuel cell (IT-SOFC) cathode $\text{Nd}_{1.95}\text{Sr}_{0.05}\text{FeO}_{4+\delta}$ was prepared by combustion method & sintered at 1273 K for 4 h. It is mixed ionic-electronic conducting (MIEC) oxides and has $K_2\text{NiF}_4$ -type structure. The prepared samples were characterized using X-ray powder diffraction (XRD), microhardness testing and four-probe dc conductivity. A close scrutiny of XRD reveals that the prepared solid solutions of cathode sintered at 1273 K are single-phase and the d -values matches well with the standard JCPDS data corresponding to pure Nd_2FeO_4 with small deviation. The dc conductivity results showed the conductivity is increased after Sr doping.

Keywords: Intermediate temperature solid oxide fuel cell, mixed ionic-electronic conductor (MIEC), combustion method, $K_2\text{NiF}_4$ -type structure, cathode.

Introduction

The solid oxide fuel cells (SOFCs) is a electrochemical device, produce electricity and heat directly from gaseous fuels through an oxidation process with high efficiency and low pollution. The operating temperature of SOFC is high (800°C – 1000°C). It is desirable to lower the operating temperature of solid oxide fuel cells from 1000°C to below 800°C. The lower operating temperature would solve various problems related to the high temperature operation, like densification of electrodes, formation of an insulating layer at the electrode/electrolyte interface by interdiffusion, and crack formation from stress caused by large differences within the thermal expansion coefficients of the cell components. Besides, low operating temperature will extend the material selection range for cell components such as the interconnect and cell housing. The reduction of operation temperature is often achieved either by thinning the electrolyte layer, or by using highly conductive electrolytes [1-5].

When a cell is operated at temperatures below 800°C with thinning the electrolytes, the cathode becomes critical for the fuel cell performance since the activation energy for cathodic reaction is very large. Therefore, many efforts are focused on decreasing the operating temperature without lowering the cell efficiency. The most promising cathode materials are mixed ionic electronic conducting (MIEC) oxides. Most of the studies on MIEC cathodes have focused on perovskites [6,7].

Now a days, a $K_2\text{NiF}_4$ family with a perovskite-related structure, has attracted more attention for use as MIEC cathodes due to favourable characteristics. The MIECs with $K_2\text{NiF}_4$ -type oxides structure exhibit anisotropic electrical transport property due to their specific crystallography characters. The structure of $K_2\text{NiF}_4$ -type oxides, as an example of A_2BO_4 (A-rare earth, alkaline-earth; B-transition metal), consists of the stacking of perovskite ABO_3 layers alternating with rock salt AO layers along the c -direction [8]. A_2BO_4 compounds exhibit high electronic conductivity. Also, possibility of forming solid solutions with mixed valence of the B-site provides good scope to tailor the physical-chemical properties. In addition, a high concentration of oxygen interstitials offers rapid oxygen transport through crystal, eventually a scope for developing a new type of MIEC cathode materials [9].

Ln_2FeO_4 (Ln= La, Ba) material is also the same kind of compound possess the same structure and contain excess oxygen. Sr doping in Ba_2FeO_4 could improve its cathodic properties. The single-phase $K_2\text{NiF}_4$ -type structures were obtained for $\text{Ba}_{2-x}\text{Sr}_x\text{FeO}_4$ over the composition range of $0.5 \leq x \leq 1.0$ for Ba [10]. Literature reveal that there has not hitherto been any report on Nd metal in the A site of $\text{Ln}_{2-x}\text{Sr}_x\text{FeO}_4$ materials. Therefore,

the study of new potential materials of the type $\text{Nd}_{2x}\text{Sr}_x\text{FeO}_4$ as cathode materials for IT-SOFCs is very interesting. In the present study, materials of the $\text{Nd}_{1.95}\text{Sr}_{0.05}\text{FeO}_{4+\delta}$ have been synthesized and characterized.

Experimental

The initial reagents neodymium acetate, strontium acetate and iron acetate, used were procured from Aldrich Chemicals (USA) with purity $> 99.9\%$. All these reagents were dried at 393 K for 24 h in order to remove the traces of moisture present. The respective requisite reagents in stoichiometric ratio were dissolved in the double distilled deionised water separately and then mixed together in a single corning flask. The homogeneous aqueous solution was then heated by using hot plate. The residue obtained was then pulverized to get them in the form of powders. The pellets of diameter and thickness 13 mm and $1\text{--}2\text{ mm}$, respectively, were obtained by uniaxially compressing ground powder at 3 tons cm^{-2} pressure with the help of Specac (UK) stainless steel die-punch and hydraulic press. The resulting pellets were initially calcined at 973 K for 4 h in an electric furnace. Subsequently, they were crushed to obtained fine powder and repelletized of diameter and thickness 9 mm and $1\text{--}2\text{ mm}$, respectively. The pellets were finally sintered at 1273 K for 4 h and allowed to cool in the furnace to room temperature.

The prepared sample was subjected to structural characterization by X-ray powder diffraction (XRD) with a PANalytical X'pert PRO (Philips, the Netherlands) instrument that employed CuK_α radiation. A curved graphite crystal was used as a monochromator. The X-ray diffraction measurement were carried out in a 2θ range from 10 to 80° with a step size and time per step of 0.020° and 5 s , respectively. The sintered densities of the sample were determined using Archimedes' principle with the help of Mettler XS105 dual range monopan balance with density kit attachment and built-in density measurement software. The micro-hardness of the sample was measured by the Vickers indentation technique (HMV2 Series Shimadzu micro-Hardness Tester, Japan).

A thin platinum film on both flat surfaces of the sintered pellet was obtained by d.c. sputtering and resulted in good ohmic contacts for d.c. electrical conductivity measurements. Prior to the conductivity measurement, the sample was spring-loaded in a ceramic cell holder (Amel, Italy) and heated to 973 K for 1 h to homogenize the charge carriers. The resistance during the cooling cycle was measured as a function of temperature using the four-probe method with a computer-controlled Keithley 6221 current source and a 2182A nanovoltmeter in delta mode. The temperature of the sample during the measurement was controlled with an accuracy of $\pm 1\text{ K}$ with a Eurotherm 2216e temperature controller. The tip of a calibrated thermocouple was kept in the vicinity of the sample to measure its actual temperature.

Result and discussion

X-ray powder diffraction

The X-ray powder diffraction (XRD) patterns of $\text{Nd}_{1.95}\text{Sr}_{0.05}\text{FeO}_{4+\delta}$ is shown in the Fig. 1. All the diffracted lines are broader than usually observed for good crystalline solids. The broadening of diffracted lines is attributed to the superfine crystallite nature of material. A careful look at the Fig. 1 revealed close matching of all the characteristic diffracted lines with the JCPDS (joint committee for powder diffraction standard) data (File No. 01-076-1876) corresponding to pure tetragonal Nd_2FeO_4 . Absence of diffracted line corresponding to either reagents or any intermediate compound confirmed the formation of single-phase tetragonal $\text{Nd}_{1.95}\text{Sr}_{0.05}\text{FeO}_{4+\delta}$ solid solution. In order to ascertain the formation of solid solution and its consequence on host lattice structure, the lattice cell constants of the samples under study were determined using Unitcell, a computer software [11]. The lattice constant a matched well with the values $a = 0.376\text{ nm}$ (of Nd_2FeO_4) but the value of lattice constant c increase. Since the ionic radii of Nd^{3+} is (11.63 nm) smaller than that of Sr^{2+} (13.1 nm) [12], partial replacement of former by latter resulted in lattice expansion of host Nd_2FeO_4 . The lattice distortion along c -axis due to partial replacement of host by smaller cation in $K_2\text{NiF}_4$ -type lattice has been reported in literature [13, 14].

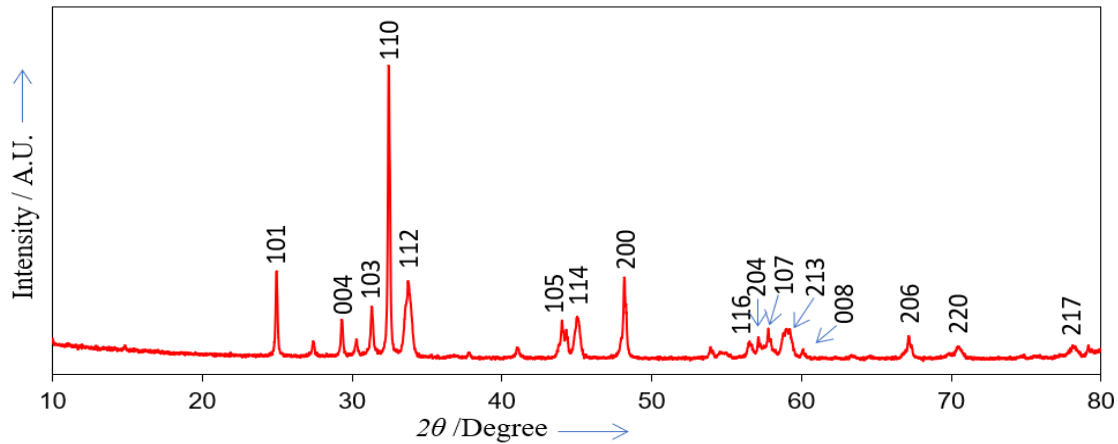


Fig. 1: The XRD patterns of Nd_{1.95}Sr_{0.05}FeO_{4+δ}

Table 1: A lattice cell constants (*a*, *c* and *v*), crystallite size (*C_s*), crystal lattice strain (*S_l*), sinter density (*ρ*) and microhardness number (*HV*) of Nd_{1.95}Sr_{0.05}FeO_{4+δ}.

Composition	<i>a</i> (nm)	<i>c</i> (nm)	<i>v</i> (nm ³)	<i>C_s</i> (nm)	<i>S_l</i> (%)	<i>ρ</i> (%)	HV
JCPDS	0.378	1.310	--	-	--	-	--
Nd _{1.95} Sr _{0.05} FeO _{4+δ}	0.376	1.315	0.186	185.3	0.12	81.6	302

The crystallite size of composition under study is 185.3 determined using X’pertHighscore plus software based on the following expression:

$$C_s = \frac{0.9\lambda}{\beta \cos \theta_B}, \quad (1)$$

where *C_s*, *λ*, and *θ_B* are thickness of crystallite, X-ray wavelength and Bragg’s angle, respectively. Here, *β* was determined by:

$$\beta^2 = \beta_m^2 - \beta_s^2, \quad (2)$$

where, *β_m* and *β_s* were the measured and the standard full width of half maxima, FWHM, of diffracted line, respectively. The *β_s* was estimated from the XRD pattern obtained by running the experiment on a standard silicon sample provided by PANalytical, Netherlands.

The sintered density (*ρ*) of the Nd_{1.95}Sr_{0.05}FeO_{4+δ} determined using the Archimedes principle, and it is 81.6. The hardness number (*HV*) of the given sample is 302.

dc conductivity

The variation of dc conductivity with temperature of the Nd_{1.95}Sr_{0.05}FeO_{4+δ} under study is shown in Fig. 2. The dc conductivity taking into account electronic contribution of MIEC cathode is measured using four-probe method.

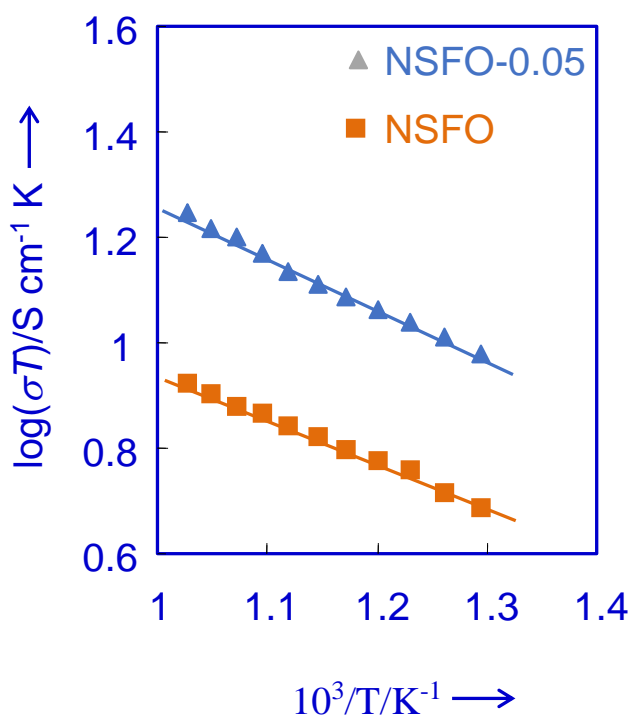


Fig. 2: Temperature Dependant dc conductivity of $Nd_{1.95}Sr_{0.05}FeO_{4+\delta}$

Given sample follows Arrhenius law within the entire measuring temperature range as follows,

$$\sigma T = (\sigma T)_0 \exp\left(\frac{-E_a}{kT}\right), \quad (3)$$

where $(\sigma T)_0$, k , T and E_a are pre-exponential factor, Boltzmann constant, absolute temperature and activation energy, respectively. It is clear from the figure that, the conductivity increases due to Sr content in the sample.

Conclusions

The $Nd_{1.65}Sr_{0.35}NiO_{4+\delta}$ solid solution prepared by combustion synthesis. XRD confirms the formation of single phase $Nd_{1.95}Sr_{0.05}FeO_{4+\delta}$. It is crystallizing with the tetragonal K_2NiF_4 -type structure. Temperature dependent dc conductivity follows the Arrhenius law. Since the Sr-doped $NdFeO_4$ is less attended as a cathode for IT-SOFC, therefore, $Nd_{1.95}Sr_{0.05}FeO_{4+\delta}$ may be a potential candidate for IT-SOFC cathode.

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