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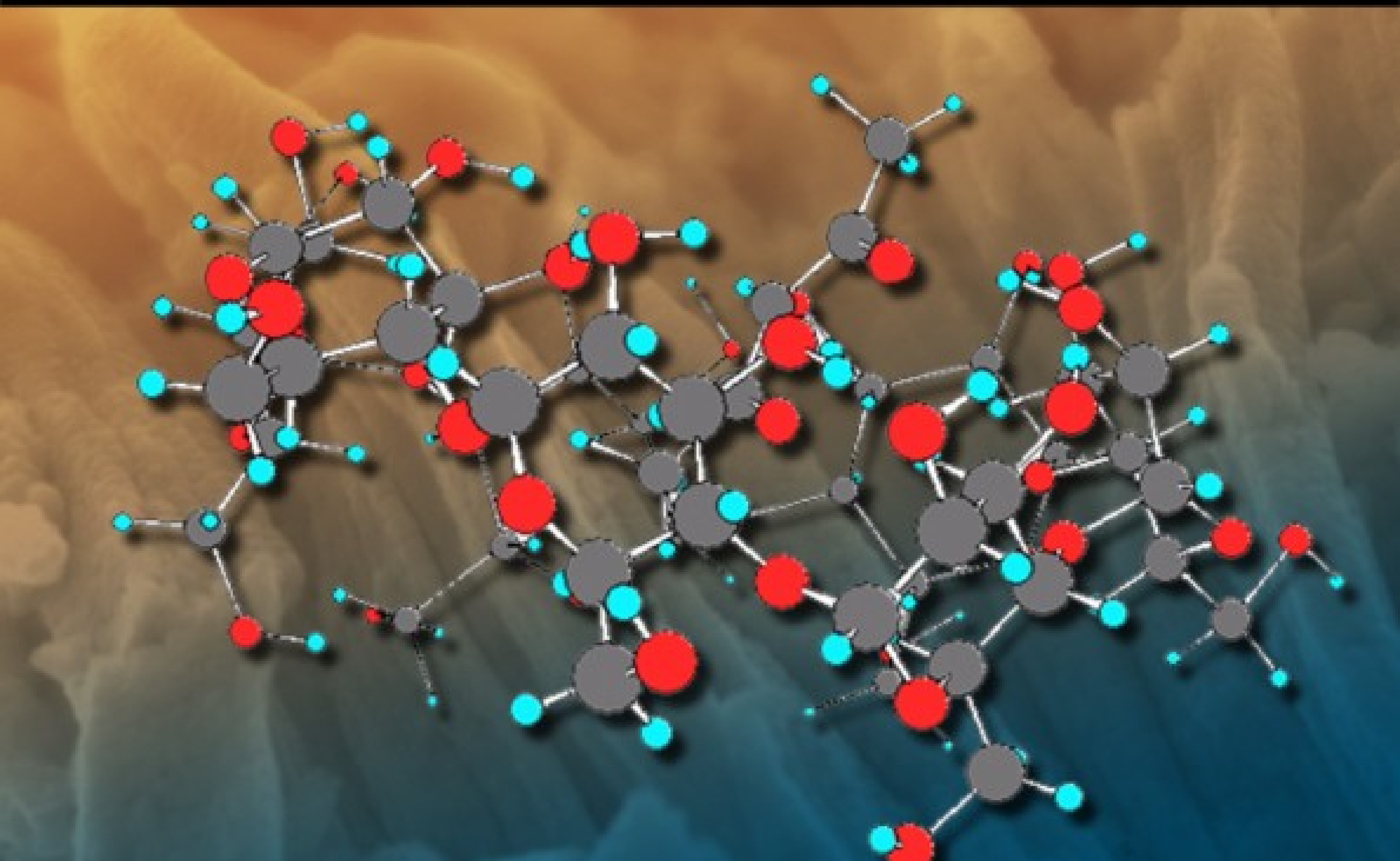

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Synthesis Characterization and Humidity Sensing Properties of Sol-gel Derived Novel Nanomaterials of $LaSr_xFe_{1-x}O_{3-\delta}$

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Abstract: $LaSr_xFe_{1-x}O_{3-\delta}$ (x=0, 0.2, 0.4, 0.6, 0.8, 1) were prepared by sol-gel method. The compounds were sintered at 700 °C for 6 hrs and were subjected to DC resistance measurements at RH 5-98 % and temperature dependent studies. The change in surface conductivity as a function of applied field was measured using picoammeter (Keithely-6485). Among the various mole ratios of compounds LSF-4 shows the highest sensitivity factor of 2.606×10^4 and LSF-1 shows the lowest sensitivity factor 0.426×10^2. Response and recovery times were measured. The compounds were characterized by X-ray diffraction, FT-IR and scanning electron microscopy (SEM) studies were employed to study the structural phases, vibrational frequencies, surface morphology of the highest humidity sensing compounds. *Copyright © 2012 IFSA.*

Keywords: Mixed metal oxides, Sensors, Perovskites, Ferrite, Humidity.

1. Introduction

Chemical sensors have attracted considerable attention in past decades due to their wide applications in air quality control, environmental protection and health care as well as security [1-3]. Many efforts have been taken in the investigations of high performance chemical sensors with high sensitivity, rapid speed, reproducibility, durability and broad range operation. Thus researchers have done to develop new materials and devices [4, 5] for example adding catalyst [6], doping metals [7] and metal oxides [8].

Humidity sensors are in great demand for quality control of production process and products a wide range of industries, such as production of electronic devices, precision instruments, textiles and foodstuffs [9] and also in many domestic applications, such as smart control of living environmental in a building. A wide variety of materials have been studied and used as sensing elements in a humidity measurement device. Amongst them ceramic oxides have shown advantages terms of thermal, physical and chemical stability and mechanical strength [10-12]. The desirable characteristics of humidity sensors are high sensitivity, chemical and thermal stability, no hysteresis, low cost and long life.

The ceramic oxides crystallize in different structures e.g. Rock salt, Wurtzite, Spinel, Zinc blende, Rutile, Fluorite, Coundum, Perovskite etc., Among them Perovskite type oxides (general formula ABO_3) have attracted much attention in recent years, because their electronic properties and hence sensing behavior of these oxides may be modified by an appropriate substitutions of cations at the A and B sites [13]. These in the form of bulk thick or thin films are being used as multifunctional sensor i.e. sensing temperature, gas and humidity [14, 15].

In the present investigation a novel nano structured $LaSr_xFe_{1-x}O_{3-\delta}$ (x=0 to 1) which was prepared by sol-gel method reveals both excellent humidity and thermal sensitivity properties. The novelty of this material is achieved by preparing the mixed metal oxide has a single phase compound which is done by sintering the samples for 8 hours at 800 °C in tubular furnace. This supported by the XRD reports. The material is characterized by SEM, XRD, and IR. The humidity dependent electrical properties of Perovskite oxide $LaSr_xFe_{1-x}O_{3-\delta}$ have been studied by preparing different mole ratios of $LaSr_xFe_{1-x}O_{3-\delta}$ by altering the addition of Fe^{3+} with Sr^{2+}. It is reported that this material is considered to be a potential candidate for oxygen separation membranes as the material reported interesting electrical properties. It has been found in the humidity sensing studies that the material shows high sensitive factor by the substitution of Fe^{3+} with Sr^{2+} due to the change in the surface morphology of the material. Electrical conductance can occur through internal surface layers of adsorbed water [16], the tiny pores can fill with water as determined by the Kelvin equation [17] leading to electrolytic conduction through the moisture or the conductance of the semiconductor itself can change due to an interaction of its surface energy states with hydroxyl radicals, especially at the grain boundaries [18, 19].

2. Experimental Procedure

2.1. Sample Preparation

Sol-gel method: A known amount of citric acid and ethylene glycol (AR) were mixed well by keeping it over a magnetic stirrer for 10 minutes around 150 °C and to this the dissolved nitrate solutions of $La(NO_3)_3$, $Fe(NO_3)_3$ and $Sr(NO_3)_2$ of appropriate mole ratios were added and stirred for 30 minutes and the mixture was heated to 150 °C till powder form is obtained.

The resulting LSF compounds were compacted to pellet at a pressure of 4 ton/sq.inch. The diameter of the pellet is 13 mm and the thickness is 2 mm. These solid pellets were sintered at 700 °C for 6 hrs in ambient air atmosphere. The samples were cooled down to room temperature at the natural cooling rate of the furnace. As the concentration of strontium increased at particular level was shown to enhance the sensitivity of humidity sensors. Since our interest is to prepare a nanomaterial the compounds were synthesized specifically by sol-gel method.

2.2. Characterization and Humidity Sensing Studies

Controlled humidity environments of relative humidity 5, 31, 51, 79 and 98 % were achieved by using anhydrous P_2O_5, saturated aqueous solution of $CaCl_2.6H_2O$, $Ca(NO_3)_2.4H_2O$, NH_4Cl and $CuSO_4.5H_2O$ in a closed desiccators at an ambient temperature of 298 K. Prior to the saturation of the pellets in the above buffers, the pellets were heated at 393K for 12 hrs to remove the adsorbed water. A degassed chamber of about 200 cm^3 was used for evaluating the response and recovery characteristics. This chamber has a provision for two-way inlet, one for transpiring the dry air and the other for moist air from a wet candle. Air drying was accomplished by transpiring the air stream through drying columns packed with anhydrous $CaCl_2$ and dry P_2O_5 connected in series. The resistance measurements in the dry air as well as in moist air alternatively helped to establish the response and recovery time of the compounds.

The DC electrical resistance at different relative humidity levels of the samples in the form of pellets was determined by a two- probe method as the present work is to measure the changes in surface conductivity as a function of applied field and current. The electrical contacts were made on the surface of the pellet by means of two thin copper wires affixed with silver paint. Given the high resistivity of the materials under investigation, the potential inaccuracy due to contact resistance is assumed negligible. The pellet was inserted in the middle of the Pyrex tube of 5cm diameter on which kanthal wire was uniformly wounded externally. The kanthal wire ends were connected to avarian to vary the temperature and a copper –constantan thermocouple kept at the pellet was used to measure the temperature of the sample. The electrodes were connected to DC power supply and the Keithley 6485 picoammeter in series. The temperature dependent conductance experiments in the temperature range of 120- 300 °C under ambient conditions were carried out to determine the activation energies for electrical conduction of the samples using linearised form of the expression

$$I=Io\ exp^{-Ea/kT}, \quad (1)$$

where I is the current, E_a is the activation energy, k is the Boltzmann constant and T is the temperature.

The structural studies were carried out using a Philips X'pert diffractometer for 2θ values ranging from 10 to 80° using CuKα radiation at λ =1.54 A°. The Fourier transform infrared (FT-IR) spectra were recorded with Perkin – Elmer spectrometer using KBr pellets whose thickness was about 1.3 mm. Each spectrum was collected at room temperature under the atmospheric pressure. The samples were dispersed in spectroscopic grade KBr pellets and were scanned in the range of 4000-400 cm^{-1}.

The surface morphology of the samples was observed on a JSM -6360 SEM analyzer operating at an accelerating voltage of 15 kV using gold coated samples.

3. Results and Discussion

3.1. X-Ray Diffraction Studies

The powder XRD patterns of LSF 1 to 6 compounds (Fig. 1) showed the characteristic peaks corresponding to $LaSr_xFe_{1-x}O_{3-\delta}$ confirming the absence of impurities and presence of mixed oxide intermediate peaks of new phases in LSF-2 to LSF-5. The XRD pattern of LSF-1 compound (JCPDS- 75-0541) data corresponds to pure lanthanum ferrite which is cubic and LSF-6 which is also cubic confirmed with JCPDS data's. The XRD pattern of compounds LSF-2 to LSF-5 were studied by comparing LSF-1 and LSF-6 the uniqueness of these XRD patterns is a proof that the compounds LSF-2 to LSF-5 is of novel.

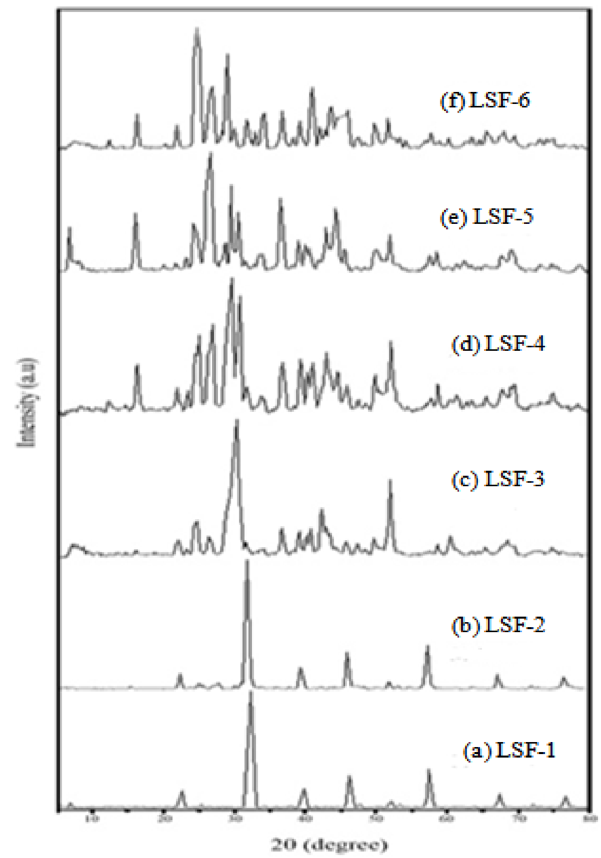

Fig. 1. XRD patterns of LSF-1 to LSF-6 compounds.

3.2. FT-IR Spectroscopy

The FT-IR spectra of LSF-1 to LSF-6 nano compounds (Fig. 2) showed spectral bands from 1000 to 400 cm^{-1} corresponds to metal oxide stretching vibrations of La-O, Sr-O and Fe-O bonds present in the compound. Since these nano compounds LSF-1 to LSF-6 is of novel they exist as overlapping peaks of La-O, Sr-O and Fe-O. The common broad band near 3448 and 1650 cm^{-1}observed were assigned to $\upsilon(HO^-)$ and $\delta(H_2O)$ [20, 21]. The FT-IR spectra of LSF-4 compound has broader band around 3400 cm^{-1} and 1600 cm^{-1} indicating more adsorption capacity for water than other compounds.

3.3. Scanning Electron Microscopy (SEM)

Fig. 3 (a-d) shows the intergranular porous structure of the sample material qualitatively. The micrographs of LSF-1and LSF-6 (Fig. 3 a, b) is of the pure lanthanum ferrite with the particle size 1 µm and pure lanthanum strontium oxide with the particle size 20 µm, which doesn't show much of porosity. The LSF-4 and LSF-5 (Fig. 3 c, d) is of the mixed metal oxides of lanthanum iron and strontium oxide which is formed as a compound by the sol-gel method. The particle size of LSF-4 is 130-180 nm and that of LSF-5 is 165-240 nm. On comparing the micrographs LSF -4 shows a larger grain size and decrease in the particle size than LSF-1, LSF-5 and LSF-6. The LSF-4 contains 0.4 moles of Fe^{3+} and 0.6 mole Sr^{2+} and LSF-5 contains 0.2 mole of Fe^{3+} and 0.8 mole of Sr^{2+}. When compared with LSF-1, LSF-5 and LSF-6 with LSF-4 shows porous nature of the compound. The morphology is well defined in the LSF-4 than inLSF-1, LSF-5 and LSF-6. The addition of 0.8 moles of strontium in the lanthanum ferrite matrix of LSF-5 reduces the grain size of the particles with the formation of intergranular pores that leads to micro porosity in addition to the presence of mesopores. Furthermore, it is observed the addition of 0.6 moles of strontium reduces the grain size and increases the pore size in LSF-4. The well developed porosity in the nanostructure LSF-4 is very important in humidity sensing studies.

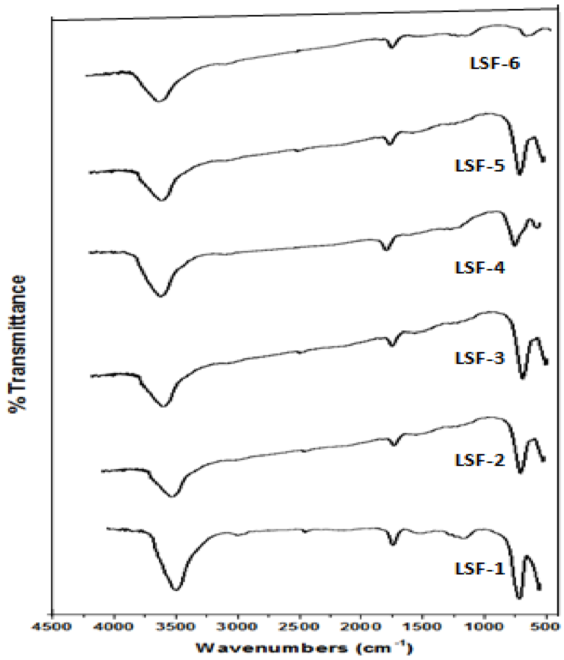

Fig. 2. FT-IR spectra of LSF-1 to LSF-6 compounds.

3.5. Humidity Measurements

In humidity sensing measurements, all the LSF compounds showed a decrease in resistance with an increase in % RH. The resistance changes in porous Perovskite type oxides with increasing of the humidity level occur because of adsorption and capillary condensation of water. The optimum concentration of x=0.4 mole ratio shows good sensor towards humidity. The LSF-4 compound showed a highest humidity sensitivity of 2606 with a resistance of 1.48×10^{10} Ω at RH 5 % and 5.68×10^{6} Ω at RH 98 %. At low humidity levels, chemisorptions takes place, leading to formation of free surface hydroxyls with the charge transport occurring by the hopping mechanism [22]. While at high humidity levels, water is physisorbed on the top of the chemisorbed layer. As a result, the condensation of water in the capillary like pores leads to a liquid like layer leading to electrolytic conduction. The increase in porosity as evidenced from SEM images confirms the presence of more sites for water adsorption in LSF-4 compound. The SEM micrographs also reveal that the LSF-4 compound produces fine grains with maximum porosity compared with the other, indicating that the smaller the grain size, the higher the surface energy and the adsorption capacity. The sintered porous semiconductor has a large internal surface area for the adsorption of water vapor. The sensitivity of the compounds with various molar ratios of LSF-1 to LSF-6 is shown in Table 1.

Further the coordination of water molecule to Fe ions in LSF-1 to LSF-6 compounds increase the acidity thereby release of H^{+}. Good linearity in the log R versus RH % plot is an important criterion for good humidity sensitivity material (see Fig. 4). The results suggest that the more linear the plot, the better the response, recovery and sensitivity of the material. The LSF-1with the humidity sensitivity factor of 43 has a resistance of 1.03×10^{10} Ω at RH 5 % and 2.42×10^{8} Ω at RH 98 %. The LSF-6 with the humidity sensitivity factor of 438 has a resistance of 5.48×10^{10} Ω at RH 5 % and 1.25×10^{8} Ω at RH 98 % due to fewer sites for water adsorption on the ceramic surface compared to that of LSF-4 compound.

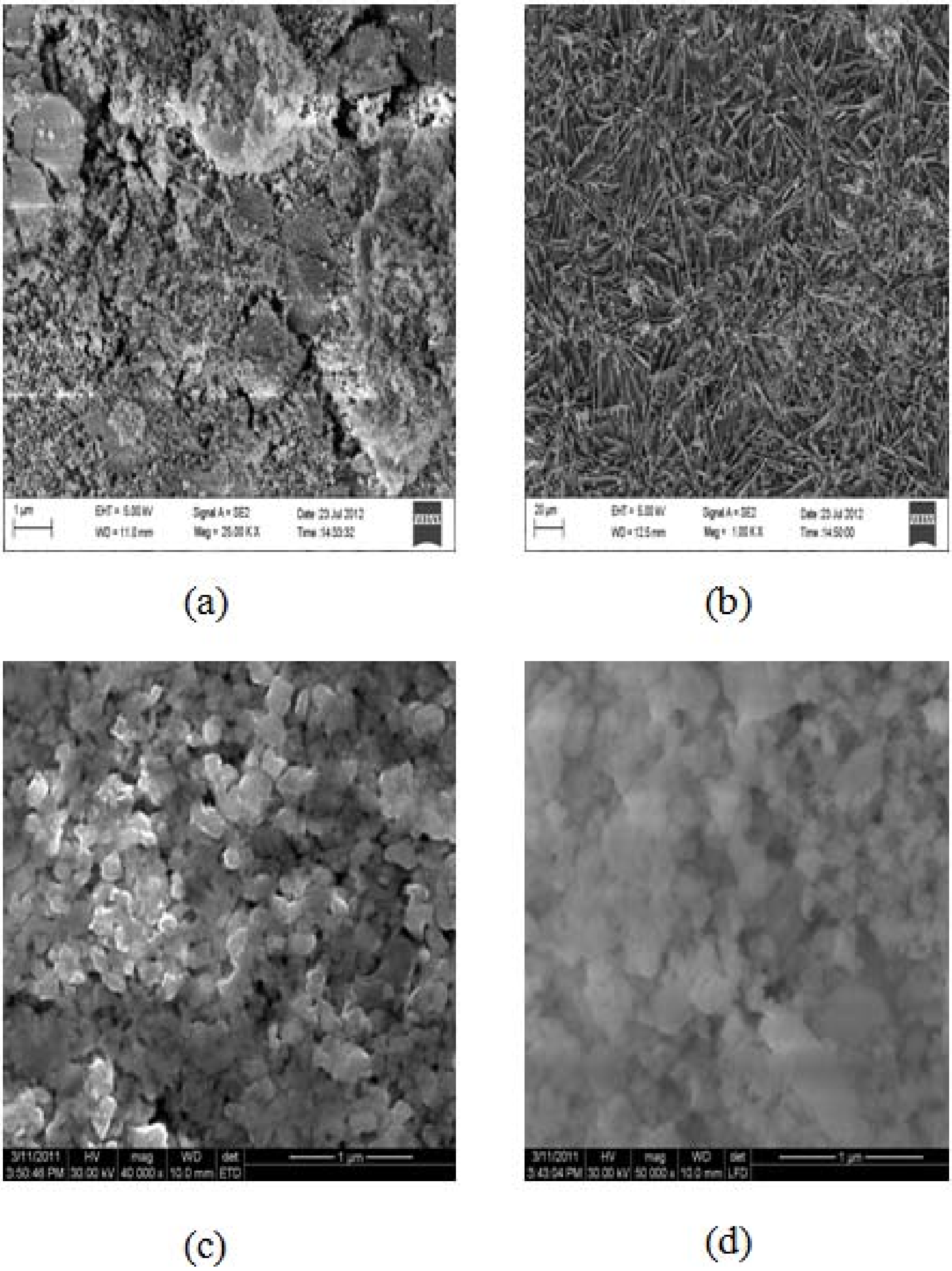

Fig. 3. SEM micrographs of a) LSF-1; b) LSF- 6; c) LSF-4, and d) LSF-5.

Table 1. Resistance at RH 5 % and RH 98 %, Sensitivity factor, Energy of activation of LSF compounds.

Sample Codes	**R(Ω), at RH 5%**	**R(Ω), at RH 98%**	**S_f ($R_{RH\ 5\%}/R_{RH\ 98\%}$)**	**E_a**
LSF-1	1.03×10^{10}	2.42×10^{8}	**42.6**	**0.094**
LSF-2	4.20×10^{10}	4.72×10^{7}	**889.8**	**0.051**
LSF-3	3.47×10^{11}	2.98×10^{8}	**1164.4**	**0.043**
LSF-4	1.48×10^{10}	5.68×10^{6}	**2605.6**	**0.034**
LSF-5	4.08×10^{10}	4.44×10^{7}	**918.9**	**0.049**
LSF-6	5.48×10^{10}	1.25×10^{8}	**438.4**	**0.040**

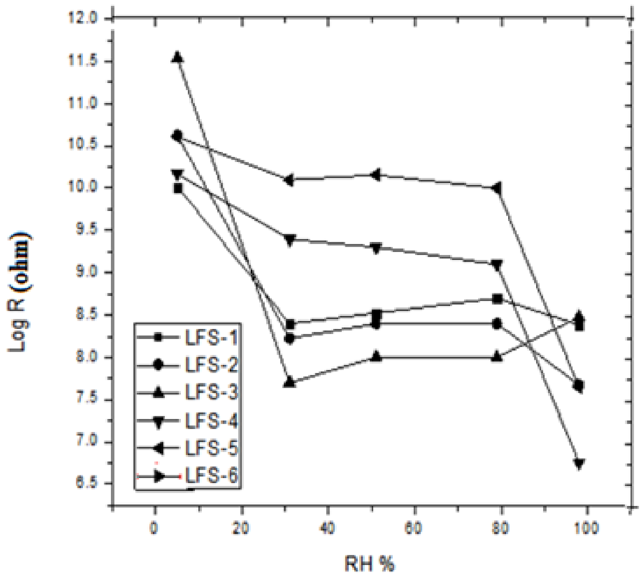

Fig. 4. Plots of log R vs. RH% for LSF-1 to LSF-6 compounds.

3.6. Temperature Dependant Studies

The electrical conductance measurements of LSF-1 to LSF-6 compounds at room temperature prior to relative humidity measurements signified that the current increased linearly with the applied voltage, indicating the ohmic contact of the electrodes. The temperature dependence of the electrical conductance carried out in the temperature range 120 °C to 350 °C suggested that the current (I) increased with an increase in temperature (T). The activation energies calculated from the temperature dependence of conductance data are also shown in Table 1. The activation energy for electrical conduction in polycrystalline materials generally involves the combination of the energy required to raise the carriers from the dominant levels to their corresponding transport bands and the energy required to create the carriers in the dominant levels [23]. The low activation energy of LSF-4 compound predicts that the small polaron conduction dominates in the studied temperature range.

3.6. Response and Recovery Characteristics

The response and recovery time obtained plots of Log R vs. Time (Fig. 5.) for LSF-4 were found to be 190 s and 80 s, respectively. The longer time taken for the restoration of the resistance to that in dry air could be understood by the fact that these experiments are conducted at 25 °C at which temperature the desorption kinetics is expected to be slow thus evidencing a surface controlled phenomena.

The response and recovery of the LSF-4 compound was further supported by the stability of the compound which is obtained by plotting Resistance vs. Days. From Fig. 6 it was found that there is no much variation in the resistance even after studying the sample after two to three months duration. This accounts for the stability of the LSF-4 compound.

Conclusion

The LSF ($LaSr_xFe_{1-x}O_{3-\delta}$) compounds with different mole ratios x= 0, 0.2, 0.4, 0.6, 0.8, 1.0 were prepared by sol-gel method and their purity was confirmed by XRD analysis. The intensity of XRD peaks shows a variation from LSF-2 to LSF-5 due to the presence of La^{3+}, Fe^{3+} and Sr^{2+}. The novelty of the compound has been proved by the XRD patterns which is different from the existing phases. The morphology of the compounds was studied by the SEM micrographs. FT-IR study showed the characteristic metal oxide vibrational frequencies. The LSF-4 ($LaSr_{0.6}Fe_{0.4}O_{3-\delta}$) compound showed the highest humidity sensitivity factor of 2.606×10^4. Temperature dependent studies showed the low activation energy of the entire compound confirming the involvement of small polaron hopping mechanism in the conduction. The good response and recovery time of LSF-4 compound and its highest sensitivity factor might be a promising humidity sensing material for practical application.

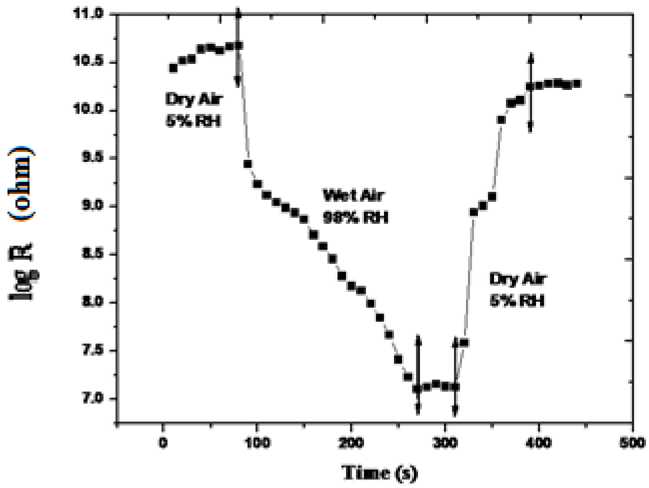

Fig. 5. Response and Recovery plots of LSF-4 compound.

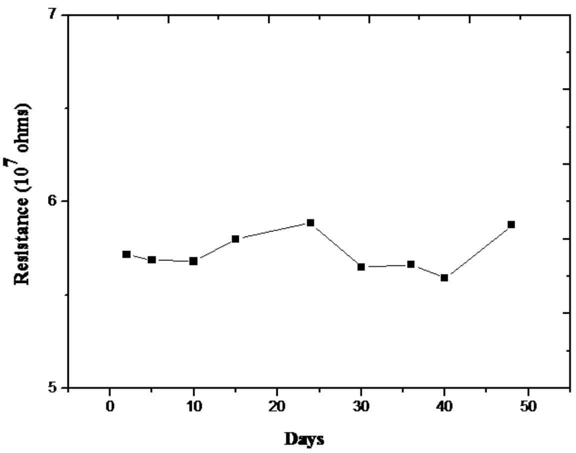

Fig. 6. Stability plot of LSF-4 compound.

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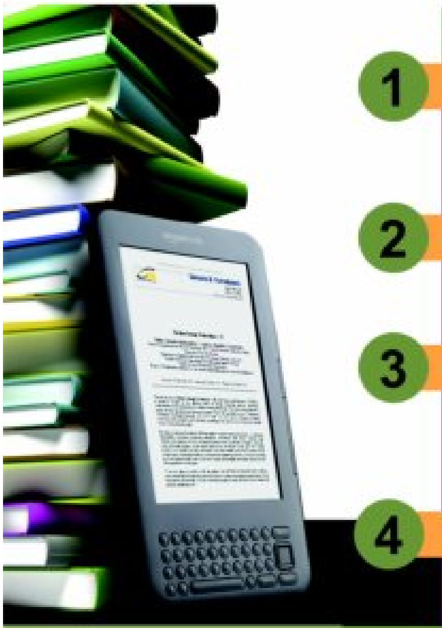

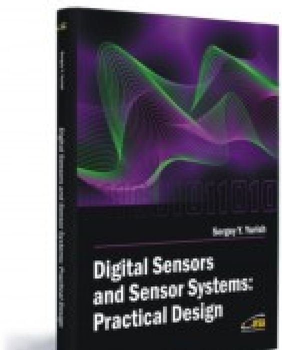
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Digital Sensors and Sensor Systems: Practical Design

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
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