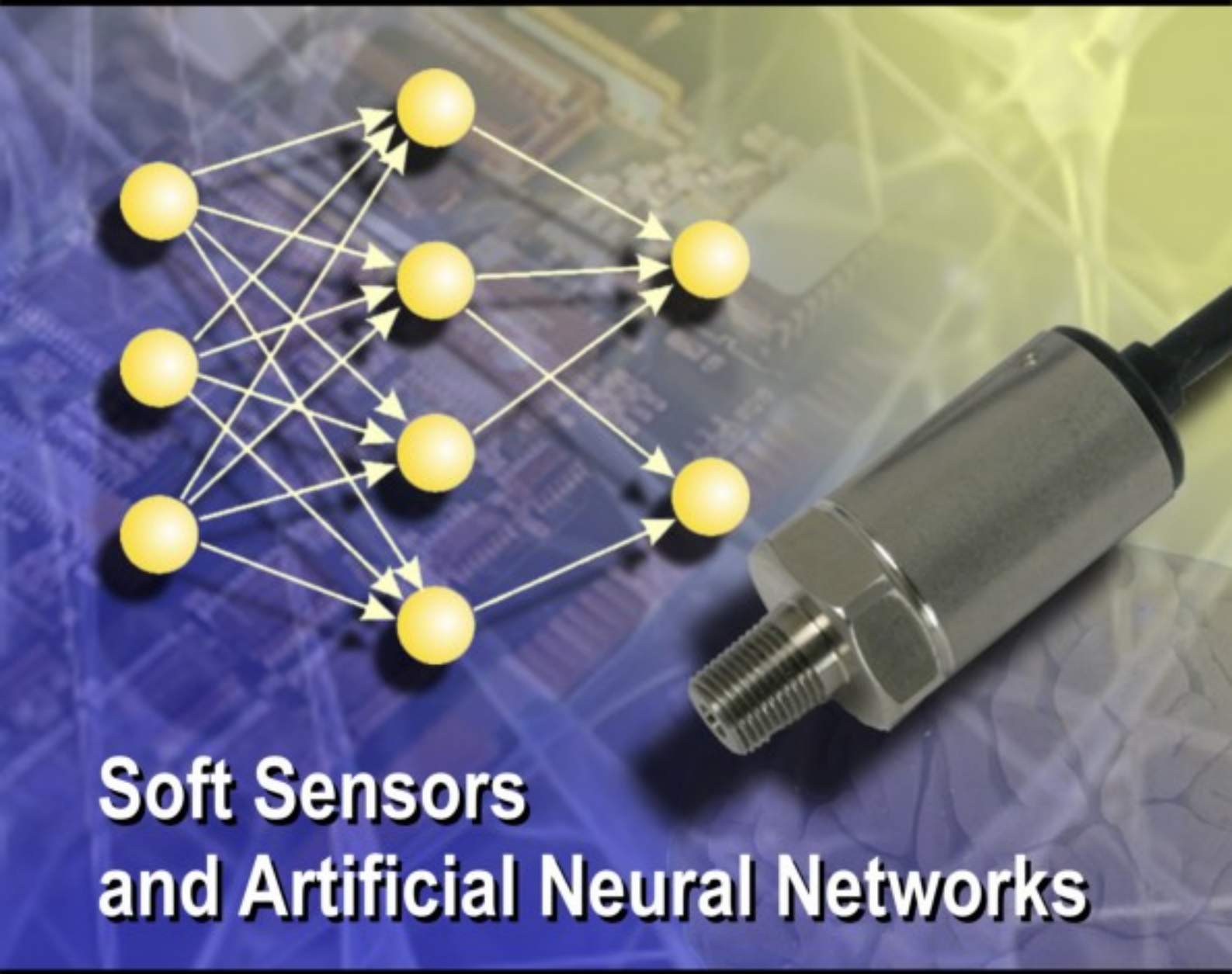
ISSN 1726-5479

SENSORS & TRANSDUCERS

vol. 97
10/08



Soft Sensors and Artificial Neural Networks

International Frequency Sensor Association Publishing

IFSA

Sensors & Transducers

Volume 97
Issue 10
October 2008

www.sensorsportal.com

ISSN 1726-5479

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Sensors & Transducers Journal (ISSN 1726-5479) is a peer review international journal published monthly online by International Frequency Sensor Association (IFSA).
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Sensors & Transducers Journal

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Issue 10
October 2008

www.sensorsportal.com

ISSN 1726-5479

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Sensors & Transducers Journal, Vol. 97, Issue 10, October 2008, pp. 74-79

www.sensorsportal.com

Sensors & Transducers

ISSN 1726-5479
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Humidity Sensor Studies on $Dy_{1-x}Sr_xCrO_3$ ($0 \leq x \leq 0.1$) Synthesized by Microwave Assisted Combustion Method

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Received: 8 September 2008 /Accepted: 22 October 2008 /Published: 31 October 2008

Abstract: The perovskite oxides, $Dy_{1-x}Sr_xCrO_3$ ($0 \leq x \leq 0.1$) were synthesized by a novel microwave assisted combustion route and characterized by X-ray diffraction. X-ray pattern of $Dy_{0.9}Sr_{0.1}CrO_3$ showed the presence of small amounts of $SrCrO_4$ impurity. The perovskite oxides were tested as candidate materials for humidity sensing by measuring the variation in their DC resistance as a function of relative humidity in the range of 5-98 % at 25°C under static conditions. The sensitivity factor, S_f ($R_{5\%}/R_{98\%}$) for $DyCrO_3$, $Dy_{0.95}Sr_{0.05}CrO_3$ and $Dy_{0.90}Sr_{0.10}CrO_3$ were calculated to be 218, 3609 and 6364 respectively. The response and recovery characteristics were studied for $Dy_{0.90}Sr_{0.10}CrO_3$, which exhibited good linearity in log R vs. RH plots and narrow hysteresis loop in adsorption-desorption investigations. *Copyright © 2008 IFSA.*

Keywords: $DyCrO_3$, $SrCrO_3$, Electrical properties, Humidity sensor

1. Introduction

Porous ceramic oxides are being investigated as candidate materials for humidity sensing due to their ideal structural and mechanical properties [1, 2]. Various synthetic routes besides conventional solid state route have also been developed [3-5] to effect the synthesis of such porous metal oxides. The variation of resistance or capacitance when water vapor is chemically or physically adsorbed on the surface results in the sensor signal. In general, humidity sensors operating at room temperature

involves H^+ conduction and hence complicated impedance measurements are needed to measure the sensor signal. However, room temperature humidity sensors operating in DC mode are also reported [6, 7], which are advantageous due to the simple instrumentation involved. The application of microwave energy has proved to be a major enabling tool for many chemical applications requiring energy input. A few techniques for microwave assisted solid phase peptide synthesis have been developed [8] that allows for enhanced deportation and coupling reactions. The experiments performed a designed experiment to determine if particular levels of selected factors would be beneficial in the optimization of a new microwave-assisted method of extraction and derivation of a substance. The $ACrO_3$ (A = large cation) perovskite oxides are quite stable both in oxidizing and reducing atmospheres and can be readily substituted on the '*A*' and '*Cr*' sites with alkaline earth and transition metal elements respectively [9], which permits interesting modifications in their electronic as well as their catalytic properties. Our recent investigation [7] on $RECrO_3$ (RE = rare earth) systems indicated that these systems, when suitably doped, can be used for commercial humidity sensing due to their high sensitivity, excellent linearity and low hysteresis at room temperature. A novel microwave assisted synthesis, characterization and humidity dependent electrical conductivity studies on $Dy_{1-x}Sr_xCrO_3$ ($0 \le x \le 0.1$) system are hereby reported.

2. Experimental Procedure

The chromates were synthesized by a novel microwave assisted process from $Dy(NO_3)_3.5H_2O$, $Sr(NO_3)_2$, $Cr(NO_3)_3.9H_2O$ and NH_2CONH_2. Calculated amounts (Table 1) of the nitrates and urea were dissolved in water and dried over a hot plate. The resultant powder was compacted into pellets, placed inside a specially designed microwave cavity (Fig. 1) and then heated in a microwave oven (1200 W, 2.45 GHz frequency) for 30 min. The phase purity was checked by powder X-ray diffraction technique, recorded in a Philips powder diffractometer with Cu-Kα radiation in the range of $10^o \le 2\theta \le 70^o$ at a scanning rate of 1.2°/min. Particle size measurements were carried out by low angle laser light scattering (LALLS) method. The density of the pellets was measured by Archimedes principle. DC conductivity studies on samples in the form of cylindrical pellets at various temperatures and humidity ambient were carried out using the two-probe technique, extensively described elsewhere [10]. Given the high resistivity of the materials under investigation, the potential inaccuracy due to contact resistance is assumed negligible. The resistance measurements in dry air as well as in moist air alternatively helped to establish the recovery and response characteristics for moisture sensing.

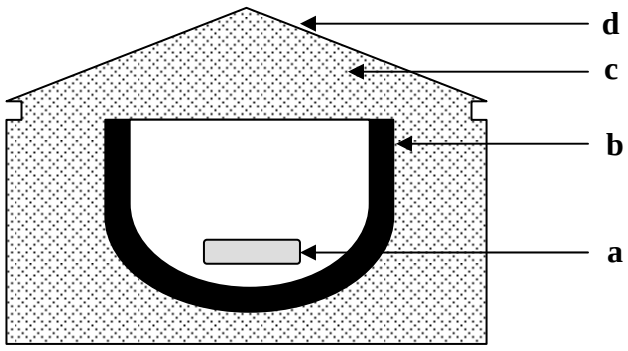


Fig. 1. Schematic of the microwave cavity:
(a) sample, (b) ceramic crucible, (c) alumina fibre, (d) SiC crucible with lid.

3. Results and Discussion

3.1. Materials Characterization and Densification

The microwave assisted synthesis of $Dy_{1-x}Sr_xCrO_3$ ($0 \le x \le 0.1$) yielded single-phase materials except for $Dy_{0.9}Sr_{0.1}CrO_3$, as identified from powder XRD (Fig. 2). A small amount of $SrCrO_4$ phase was identified as impurity in $Dy_{0.9}Sr_{0.1}CrO_3$ which also appears in significant amounts in all compositions above $x>0.1$. $DyCrO_3$, $Dy_{0.95}Sr_{0.05}CrO_3$ and $Dy_{0.90}Sr_{0.10}CrO_3$ crystallized in the orthorhombic space group, *Pbnm* [11]. The as-synthesized powders were vibromilled for 96 h, followed by vigorous stirring in iso-propanol to reduce the particle size to 10μm or less. Fine particle size is quite important for pressing the samples as pellets with reasonable density, especially in chromates where the poor sinterability and difficulties in densification are documented [12]. The milled polycrystalline samples were pressed into pellets of approximately 1 cm diameter at 300 MPa and sintered at 1000^{o}C in air at a heating rate of 2^{o}C/min. The relative density, as measured by Archimedes principle, indicated that the pellets of the strontium doped and undoped $DyCrO_3$ to be in the range of 52 – 58 %. The density increases slightly with increasing strontium concentration. The low relative density is due to the neck growth with poor densification during sintering, commonly observed in chromium oxides. Nevertheless, chromates can be densified by sintering under reduced pressure followed by annealing in air to fully oxidize the samples. But our earlier studies on mixed metal oxide systems revealed that the presence of microporous structure facilitates water occlusion in pores, enhancing the sensitivity [13] and hence no efforts were made to further densify the materials.

3.2. Electrical Conductivity

In electrical conductivity measurements, the current was found to increase linearly with the applied voltage, indicating the ohmic contacts of the electrodes. The temperature dependence of electrical conductivity was carried out in the temperature range of 100–300^{o}C. The current (I) increased exponentially with respect to temperature (T). From the temperature dependence conductivity data, *ln I* vs 1/*T* plot was made using the Arrhenius equation from which the activation energy (Ea) was determined [13]. The Ea calculated for $DyCrO_3$, $Dy_{0.95}Sr_{0.05}CrO_3$ and $Dy_{0.90}Sr_{0.10}CrO_3$ are 0.60, 0.56 and 0.37 eV respectively.

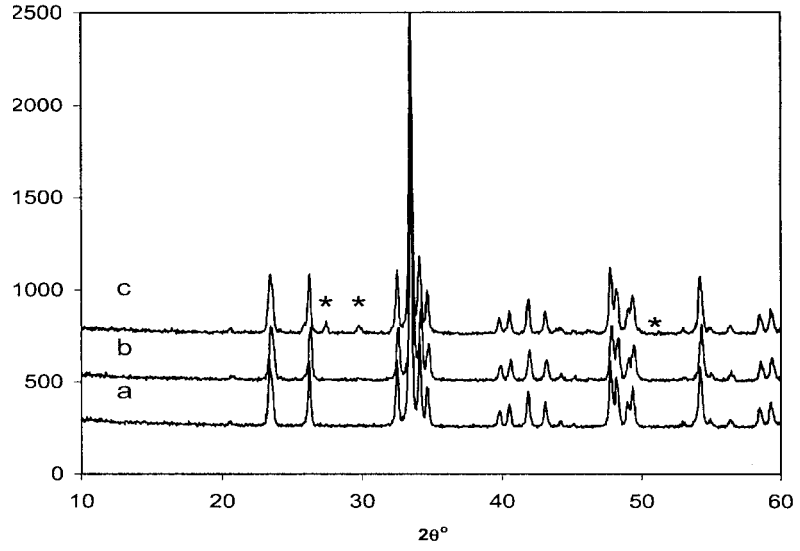


Fig. 2. Experimental XRD patterns of (a) $DyCrO_3$, (b) $Dy_{0.95}Sr_{0.05}CrO_3$ and (c) $Dy_{0.90}Sr_{0.10}CrO_3$.

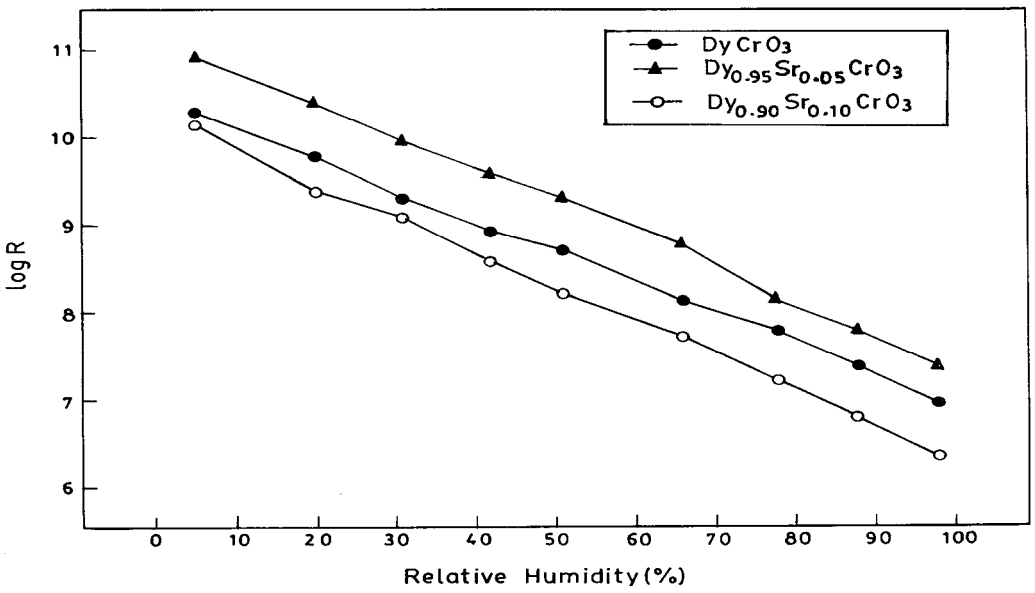


Fig. 3. RH vs log R plots at 25°C for $Dy_{1-x}Sr_xCrO_3$ (0<x<0.1) system.

3.3. Humidity Sensitivity

The results of resistance measurements as a function of relative humidity at a fixed ambient temperature of 25°C are presented in Fig. 3. As the humidity is increased, the log *R* value drops (Fig. 3) and the sensitivity of the oxides towards humidity increases (Table. 1). The resistance of both doped and undoped oxides changed by almost four orders of magnitude during the relative humidity variation between 5 to 98 % RH. The large decrease in resistance with increased relative humidity in ceramic oxides is related to the adsorption of water vapors on the grain surface and in the pores. The small particle size of the grains facilitated by the microwave assisted combustion synthesis results in more grain boundaries and pores, leading to more active sites available for condensed water to get adsorbed. The resistance of $DyCrO_3$, $Dy_{0.95}Sr_{0.05}CrO_3$ and $Dy_{0.90}Sr_{0.10}CrO_3$ decreased linearly with increasing relative humidity, which indicated the absence of any electrode charging. The sensitivity factor (S_f) was calculated from the ratio of $R_{5\%}/R_{98\%}$, where $R_{5\%}$ and $R_{98\%}$ are the DC resistances at 5 and 98 % RH respectively. The sensitivity factor (Table 1) of $DyCrO_3$, $Dy_{0.95}Sr_{0.05}CrO_3$ and $Dy_{0.90}Sr_{0.10}CrO_3$ is 218, 3609 and 6364 respectively. Thus the sensitivity factor increases with increasing 'A' site doping which involves substitution of Dy^{3+} with Sr^{2+} resulting in increasing oxygen vacancies. The systematic increase in the sensitivity factor with increasing oxygen vacancies infer that the probable contribution of the $SrCrO_4$ impurity to humidity sensitivity to be negligible. The influence of oxygen vacancies on the moisture sensitivity remains unclear and warrants further investigation.

Table 1. Mole composition and Sensitivity factor of the $Dy_{1-x}Sr_xCrO_3$ (0<x<0.1) system.

Weight (Moles)			Perovskites	Resistance (Ω)		S_f = ($R_{5\%}/R_{98\%}$)
$Dy(NO_3)_3$	$Cr(NO_3)_3$	$Sr(NO_3)_2$		$R_{5\%}$	$R_{98\%}$	
1	1	--	$DyCrO_3$	1.9×10^{10}	8.7×10^{7}	218
0.95	1	0.05	$Dy_{0.95}Sr_{0.05}CrO_3$	8.3×10^{10}	2.3×10^{7}	3609
0.90	1	0.10	$Dy_{0.90}Sr_{0.10}CrO_3$	1.4×10^{10}	2.2×10^{6}	6364

Adsorption and desorption behavior of the maximum humidity-sensitive oxide, $Dy_{0.90}Sr_{0.10}CrO_3$, were studied in the range of 5 – 98 % RH at 25 °C (Fig. 4). The sample was statically housed under each RH

condition with equal time intervals to attain equilibrium. $Dy_{0.90}Sr_{0.10}CrO_3$ exhibited narrow hysteresis loop (Fig. 4) in the entire range of study. This indicates facile adsorption and desorption of water and is a prerequisite for commercial sensors. At low humidity range, water adsorption on the oxide surface is likely the dominant factor for electrical conduction whereas at higher humidity, the microstructure dominates in determining largely the total quantity of occluded water [14]. As a result the electrical conduction is likely to be due to continuous water layers within the pores and hence the system exhibits higher sensitivity.

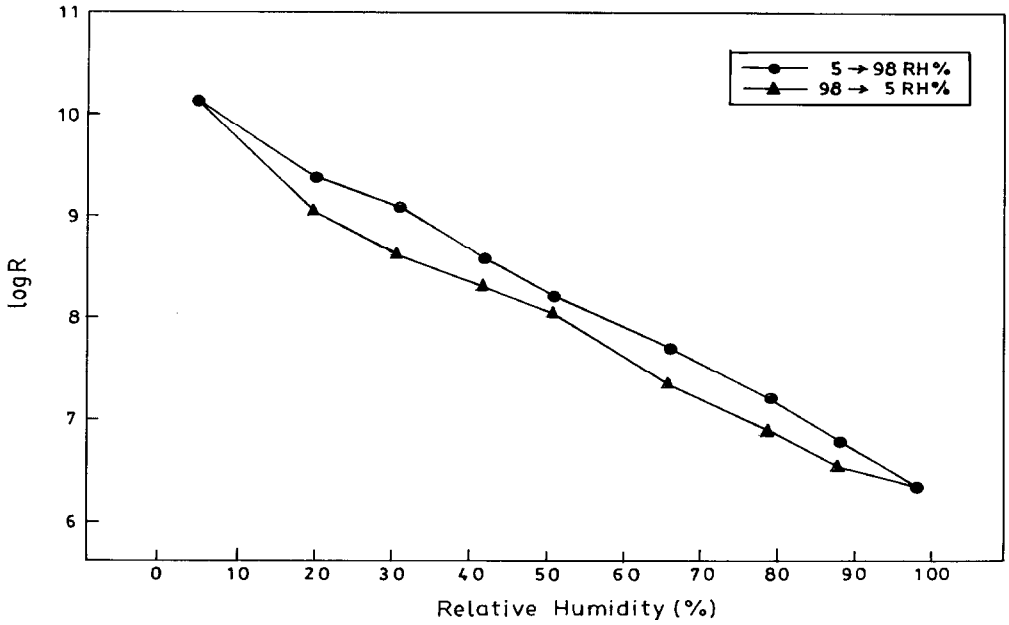


Fig. 4. Adsorption and desorption behaviour of $Dy_{0.90}Sr_{0.10}CrO_3$ at 25°C**.**

3.4. Response and Recovery Characteristics

The oxide with good sensitivity and narrow hysteresis, $Dy_{0.90}Sr_{0.10}CrO_3$ was chosen for the evaluation of response and recovery characteristics. The DC resistance in dry air as well as in moist air alternatively helped to establish the response and recovery time. The invariant resistance in dry air is in the order of 10^8 ohms about 10 min. of purging with moist air, the resistance dropped (Fig. 5) to reach a constant value of approximately 10^6 ohms. The time taken for the restoration to the original signal on passing dry air is 11 min.

4. Conclusion

Single phase $DyCrO_3$ and $Dy_{0.95}Sr_{0.05}CrO_3$ as well as $Dy_{0.90}Sr_{0.10}CrO_3$ containing small amounts of $SrCrO_4$ impurity were prepared by a novel microwave assisted combustion process and characterized by x-ray diffraction. The perovskites were fabricated as discs and studied for their sensitivity towards humidity at 25°C. $Dy_{0.90}Sr_{0.10}CrO_3$ was found to exhibit a good linearity, narrow hysteresis loop with sensitivity factor of 6364, the response and recovery time being approximately 10 and 11 min, respectively.

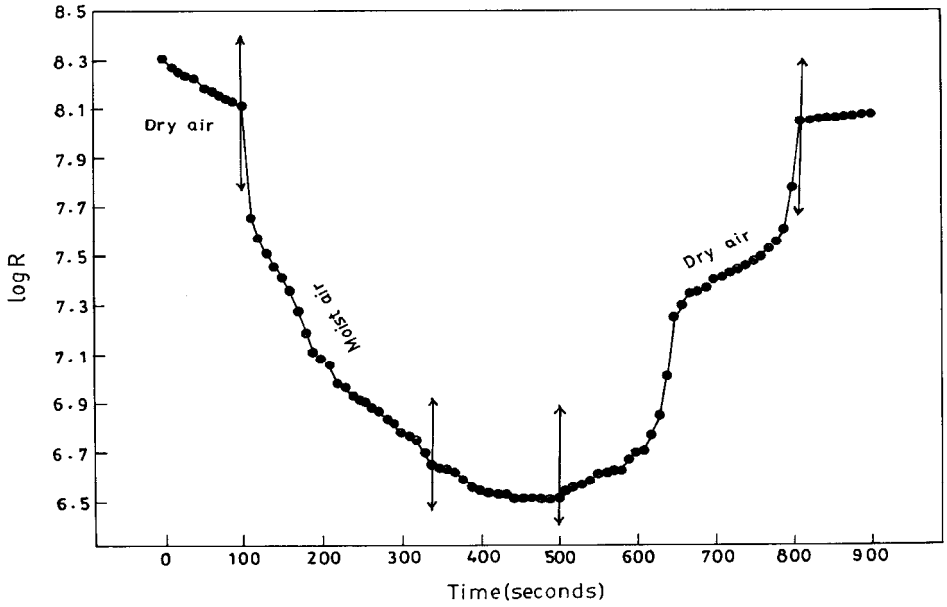


Fig. 5. Resistance vs time plot of $Dy_{0.90}Sr_{0.10}CrO_3$ exposed to dry and moist air alternatively at 25°C.

Acknowledgements

The authors are thankful to Thiru. P. Ponnambalam, Secretary and Dr. S. Rajagopalan, Principal, Nehru Memorial College for their support and encouragement. We wish to thank Prof. Dr. R. Rengasamy Centre for Advanced Studies in Botany, University of Madras and Chennai for his support and helpful discussions.

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Contacts

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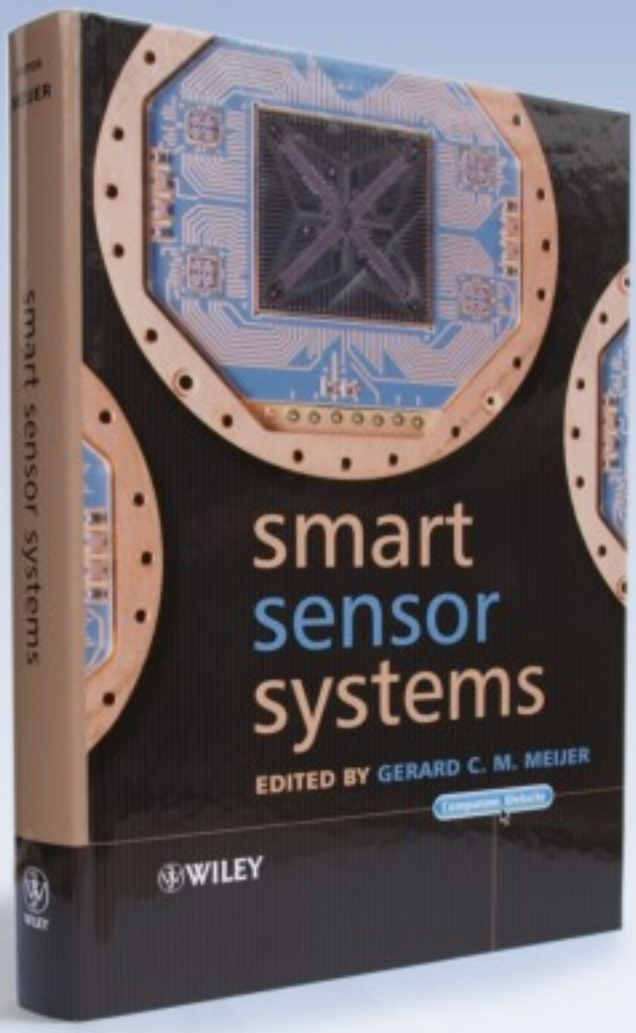
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