Synthesis and characterization of nickel manganite from different carboxylate precursors for thermistor sensors

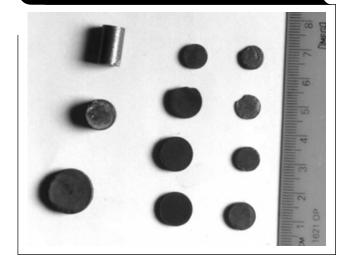
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Introduction

The thermistor sensor is a widely used temperature transducer synthesized with a ceramic-like semiconductor material. Its basic temperature-sensing mechanism is the population of charge carriers in the conduction band. Initially the thermistor was designed for use during World War II, but since then it has continuously evolved for applications such as thermometers, temperature controllers, automatic gain control, and time-delay circuits. Its number of applications is increasing rapidly, encompassing office automation equipment, air conditioners, and other domestic appliances. Recently the thermistor has come into the limelight due to its use in portable phones, car phones, and transceivers. Expanding applications in delicate communications equipment and the rapid influx of such equipment into the consumer market has forced thermistor manufacturers to consider improving the specifications. In this article the authors investigate various thermistor anomalies from a materials point of view. Figure 1 shows thermistor samples we manufactured in various sizes. The small samples are suitable for applications where speed of measurement is crucial.

The thermistor sensor offers various advantages: high sensitivity; availability in a large range of resistance values (useful from a power dissipation point of view); ability to

Figure 1. Thermistor samples



operate over a wide temperature range in a solid, liquid, or gaseous environment; adaptable size and shape for a wide variety of mechanical environments; ability to withstand electrical and mechanical stress; and low cost. At the same time, this sensor has several limitations: lack of interchangeability; poor linearity and precision; limited range; instability at high temperatures; hysteresis; and low resolution. With the advent of microprocessors and microcontrollers, most of these drawbacks are no longer a problem except for lack of interchangeability. The authors investigate remedies for the bottlenecks posed by thermistors in References 16, 17, and 18. On several occasions we observed that the thermistor-based circuit needs tuning when it is changed. The reasons for lack of interchangeability are loose manufacturing standards and manufacturers' ignorance of standard material characterization tools used to check completion of solid-state reactions (formation).

Materials used

At present the thermistor materials of interest and use are mixed metal oxides (especially spinels) of manganese, nickel, cobalt, copper, iron, and titanium. The authors of References 1-5 show an interest in transition metal manganite, $Mn_{3-x}M_xO_4$ [(0 < x < 1) and M = nickel, cobalt, etc.]. Nickel manganite, NiMn₂O₄, is a popular material for thermistors despite its poor stability at high temperature. The electrical conductivity is due to hopping between Mn³⁺ and Mn⁴⁺ ions in the octahedral sub-lattice of the spinel structure. In this article the carboxylate precursor method, used for the first time to synthesize NiMn₂O₄, is described. The different carboxylates used for precursor formation are fumarate, succinate, oxalate, tartarate, and malonate. The precursors as well as the NiMn₂O₄ obtained by thermal decomposition of these precursors have been characterized by X-ray diffraction, infrared analysis, and thermal analysis to study the formation and working.

Preparation

Different nickel manganese carboxylate precursors were prepared by using salts of carboxylic acid and metal chlorides. Following is a description of how the metal chloride solution and the precursors were prepared.

Preparation of metal chloride solution

Around 11.8854 g of NiCl₂.6H₂O (0.5M) was accurately weighed on a Mettler balance and dissolved in distilled water. Similarly, 19.79056 g of MnCl₂.4H₂O (1M) was also accurately weighed and dissolved in acidified water [to prevent formation of Mn(OH)₂]. Both salt solutions were

then mixed together to make the volume 100 ml in a standard volumetric flask. This metal chloride solution was then used for the preparation of the nickel manganese carboxylate precursors.

Preparation of nickel manganese fumarate (NMF)

Sodium fumarate was accurately weighed at 32.008 g (2M) and dissolved in distilled water; the total volume was made 100 ml in a standard flask. This solution was then heated to 80°C. The hot metal chloride solution was added drop-by-drop, with constant stirring, to this hot fumarate solution. The precipitate of nickel manganese fumarate that formed was filtered in a Buchner funnel using a Whatmann filter No. 41. It was washed with distilled water until it was free of chloride ions, then dried with diethyl ether and stored in a desiccator.

Preparation of other precursors

The other precursors—nickel manganese succinate (NMS), nickel manganese oxalate (NMO), nickel manganese tartarate (NMT), and nickel manganese malonate (NMM)—were also prepared in the same manner as described for NMF by using sodium succinate, ammonium oxalate, sodium tartarate, and sodium malonate, respectively, along with metal chloride.

Characterization

The nickel manganese carboxylate precursors were characterized by chemical analysis. The ${\rm NiMn_2O_4}$ obtained by thermal decomposition of the carboxylate precursors was also characterized by X-ray diffraction and IR analysis. The results of all the characterizations are summarized in Tables 1-9.

Chemical analysis

The percentage of nickel and manganese in the precursors was estimated by using the standard methods described in Reference 23. The elemental analysis was carried out on an AAS 201 Chemito GBC902, double-beam atomic absorption spectroscope. The wavelengths used for nickel and manganese estimation were 352.4 nm and 403.1 nm, respectively. The standard solutions prepared for nickel and manganese were in the range of 6 to 25 μ g/ml and 7 to 27 μ g/ml, respectively.

IR analysis

IR analysis of the precursors and their decomposed products was carried out on Shimadzu FTIR instrument model 8101A. The pellets used for reading spectra were prepared by mixing 1 to 2 mg of the sample with a pinch of KBr. The IR spectra in the range of 400 to 4600 $\rm cm^{-1}$ was recorded at room temperature.

Density measurement

The pycnometric density measurement of the precursors and their decomposed products was determined at room temperature with CCl₄ as the medium by using the formula p_{sample} = (weight of the sample)/(weight of the liquid displaced/density of the liquid).

Thermal analysis

The physical and chemical properties of the precursors were monitored by using thermal analysis techniques like isothermal weight loss studies, thermogravimetry analysis (TGA), and differential thermal analysis (DTA). For calculating the weight loss, precursors were accurately

weighed and placed in silica crucibles, then heated in an oven for 20 minutes at various temperature ranges until the precursors exhibited no further weight loss. TGA was done on an STA 1500 instrument in air at a heating rate of 10°C/min. DTA was recorded on an STA 1500 instrument in air at a heating rate of 10°C/min.

X-ray diffraction (XRD) analysis

X-ray diffraction of the sintered and decomposed products was carried out on Philips X-ray Diffractometer model PW 3710 with Cu $K\alpha$ radiation and nickel as a filter. The studies were carrried out to confirm the completion of solid state reaction, observe the impurity phases, and determine lattice constants, interplanar distances, octahedral and tetrahedral site radii, bond length, X-ray density, etc. The various parameters were calculated using standard values.

Formula fixation

Based on the characterization results previously described, a formula for each precursor was fixed as follows:

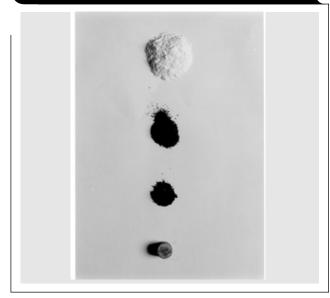
NMF: NiMn₂(C₄H₂O₄)₃.4¹/₂H₂O NMS: NiMn₂(C₄H₄O₄)₃.10H₂O NMO: NiMn₂(C₂O₄)₃.6¹/₂H₂O NMT: NiMn₂(C₄H₄O₆)₃.8H₂O NCM: NiMn₂(C₃H₂O₄)₆.3H₂O

Pellet formation

Some of the steps in thermistor manufacture are shown in Figure 2. The thermistors were fabricated by preheating, presintering, grinding, and shaping them to the desired geometry and by a final sintering at elevated temperature. The thermally decomposed product of the precursors was preheated in a silica crucible to $400^{\circ}\mathrm{C}$ to drive off

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Figure 2. Some steps in thermistor manufacture



The light-colored powder at the top is raw precursor. Below that is pre-sintered powder before and after it is finely ground and filtered. The weight loss that results is evident. Compressing the sintered precursor in a hydraulic press produces the finished thermistor.

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moisture. The preheated product was mixed thoroughly by grinding and then was compressed under 5 tons per square inch of pressure in a hydraulic press in a round die. These pellets were then heated to 900°C under a controlled temperature profile using a PID controller. The final products were disc thermistors with diameters of 1 mm (0.04 in.) to 3 mm (0.12 in.), targeting low-cost thermometry applications for domestic use.

Results and conclusion

This article presents our work on the preparation of nickel manganese carboxylates by various precursor methods; viz., nickel manganese fumarate, nickel manganese succinate, nickel manganese oxalate, nickel manganese tartarate, and nickel manganese malonate. Various characterization tools were applied to these precursors to verify the formation. The asymmetric and symmetric stretching

of carboxylate ions was seen in the range of 1550 to 1625 cm⁻¹ and 1350 to 1400 cm⁻¹, respectively, with Δv (v_{asys} - v_{sys}) separation of ~190 to 240 cm⁻¹, indicating the monodentate linkage of both carboxylates in the dianions. Thus the carboxylates coordinated to the metal as bidentate ligand via both carboxylate groups. Almost all the hydrate nickel manganese carboxylate precursors decomposed below 400°C to form NiMn₂O₄. Most of the dehydration took place below 250°C, while the decarboxylation of anhydrous precursors occurred between 250 and 400°C. XRD confirmed the formation of NiMn₂O₄. The lattice parameter values of all the samples agreed well with the reported ones. The IR data of NiMn₂O₄ showed high-frequency band v1 between 600 and 620 cm⁻¹ and low-frequency band between 450 and 460 cm⁻¹. The I-V characterization of all the samples up to 200°C with four-probe setup revealed their thermistor behavior. The low value of resistivity at room temperature was attributed to the presence of moisture.

Table 1. Infrared data of hydrated nickel manganese carboxylates

CARBOXYLATES	INFRARED DATA			cm ⁻¹		
CARBUXTLATES	∨(H ₂ O)	∨ _{asym} (0-C-0)	∨ _{sym} (0-C-0)	δ(0-C-0)	V(M-0) + V(C-C)	∨(CH=CH)
NMF	3475	1580	1390	800	590	990
NIVIF	3400	1560				
NINAO	3400	1550	1350	800	660	_
NMS			1400			
NM0	3400	1625	1360	840	495	_
NMT	3400	1575	1380	800	575	_
NCM	3400	1575	1400	840	565	_
INCIVI					530	

Table 2. Chemical analysis, total weight loss and density of hydrated nickel manganese carboxylates

		CHEMICAL	ANALYSIS		TOTAL MELO	NIT LOCE (N/)	DENCITY
CARBOXYLATES	NICKEL (%)		MANGANESE (%)		TOTAL WEIGHT LOSS (%)		DENSITY
	OBS.	CALC.	OBS.	CALC.	OBS.	CALC.	- (gcm ⁻³)
NMF	9.4315	9.9176	17.855	18.5663	60.62	60.70	1.7929
NMS	10.8211	8.4217	14.28	15.7657	66.63	66.65	2.0192
NM0	9.8526	10.6768	20.8	19.98	57.46	57.38	2.1585
NMT	6.95	7.7545	15.34	14.5168	69.45	69.72	1.7304
NCM	11.00	11.1003	16.02	20.7804	56.14	56.01	1.7534

Table 3. Isothermal weight loss and TGA/DTA of nickel manganese carboxylates

	TGA (AIR)		DTA PEAKS	ISOTHERMAL WEIGHT LOSS			
CARBOXYLATES	TEMPERATURE	WT. LOSS	EXO/ENDO PEAKS	TEMPERATURE	WT. LOSS	DEMARKS	
	RANGE (°C)	(%)	(°C)	RANGE (°C)	(%)	REMARKS	
	RT-91.22	0.62		RT-100	16.80	Loss of $6^{1/2}$ H_2O	
	91.22–176.8	26.002	135.27 (endo)	100–120	6.40	Loss of 2 ¹ / ₂ H ₂ 0	
				120–140	4.66		
NMS	176.8–396.76	28.596	331.7 (exo hump)	140-250	22.24	Loop of 1 H O	
			387.44 (broad exo)	250-380	11.93	Loss of 1 H ₂ O	
	396.76-526	11.081-0.376				Decarboxylation to form NiMn ₂ O ₄	
	526-790		602.04 (exo)			Decarboxylation to form MivingO4	

Table 4. Isothermal weight loss studies of hydrated nickel manganese carboxylates

CARBOXYLATES	TEMPERATURE RANGE (°C)	WEIGHT LOSS (%)	REMARKS
	RT-120	7.61	Loss of 2 ¹ / ₂ H ₂ 0
NMF	120-140	2.69	Loss of 1 H ₂ O
INIVIF	140-250	3.21	Loss of 1 H ₂ O
	250–280	48.19	Decarboxylation to form NiMn ₂ O ₄
	RT-100	14.74	Loss of 4½ H ₂ O
NMO	100–160	1.64	Loss of ½ H ₂ O
INIVIO	160-200	4.92	Loss of 1½ H ₂ 0
	200–280	36.39	Decarboxylation to form NiMn ₂ O ₄
	RT-100	10.11	Loss of 4 H ₂ O
	100–120	2.38	Loss of 1 H ₂ O
NMT	120-180	4.17	Loss of 2 H ₂ O
INIVII	180–225	2.33	Loss of 1 H ₂ O
	225–250	3.53	Decarboxylation to form NiMn ₂ O ₄
	250-380	46.70	Decarboxylation to form Milving04
	RT-120	1.73	Loss of ½ H ₂ O
	120–170	4.90	Loss of 11/2 H ₂ 0
NMM	170–180	2.75	Loss of 1 H ₂ O
	180–260	32.65	Decarboxylation to form NiMn ₂ O ₄
	260–380	3.90	Decarboxylation to form Milving04

Table 5. X-ray diffraction data of NiMn₂O₄ (NMF)*

SR. NO.	2 θ (°)	d _{obs} (A°)	d _{calc} (A°)	HKL
1	30.25	2.9545	2.9611	220
2	35.55	2.5252	2.5252	311
3	43.20	2.0941	2.0938	400
4	53.60	1.7098	1.7096	422
5	57.05	1.6143	1.6132	333
6	62.80	1.4795	1.4805	440

^{*}Lattice parameter a = 8.39805 Structure—cubic

Table 6. X-ray diffraction data of NiMn₂O₄ (NMS)*

SR. NO.	2 θ (°)	d _{obs} (A°)	d _{calc} (A°)	HKL
1	18.33	4.8481	4.8441	111
2	30.16	2.9676	2.9664	220
3	35.54	2.5298	2.5298	311
4	37.17	2.4226	2.4221	222
5	43.20	2.0976	2.0975	400
6	53.61	1.7122	1.7127	422
7	62.78	1.4825	1.4832	440
8	75.35	1.2634	1.2649	622

^{*}Lattice parameter a = 8.39005 Structure—cubic

Table 7. XRD data of NiMn₂O₄ obtained from carboxylates (NMO)*

SR. NO.	2 θ (°)	d _{obs} (A°)	d _{calc} (A°)	HKL
1	18.36	4.8289	4.8449	111
2	30.13	2.9705	2.9705	220
3	35.49	2.5333	2.5333	311
4	37.15	2.4238	2.4254	222
5	43.14	2.1002	2.1003	400
6	53.53	1.7146	2.7151	422
7	57.23	1.6124	1.6169	333
8	62.84	1.4813	1.4853	440
9	74.15	1.2809	1.2813	533
10	75.18	1.2658	1.2666	622

^{*}Lattice parameter a = 8.397742 Structure—cubic

Table 8. X-ray diffraction data of NiMn₂O₄ (NMT)*

SR. NO.	2 θ (°)	d _{obs} (A°)	d _{calc} (A°)	HKL
1	30.15	2.9641	2.9692	220
2	35.45	2.5321	2.5321	311
3	43.25	2.0918	2.0995	400
4	53.70	1.7068	1.7143	422
5	57.10	1.6130	1.6162	333
6	62.7	1.4817	1.4845	440

^{*}Lattice parameter a = 8.38848 Structure—cubic

Table 9. X-ray diffraction data of NiMn₂O₄ (NMM)*

SR. NO).	2 θ (°)	d _{obs} (A°)	d _{calc} (A°)	HKL
1		35.53	2.5308	2.5308	311
2		37.34	2.4122	2.4230	222
3		43.37	2.0898	2.0984	400
4		57.31	1.6103	1.6154	333
5		63.01	1.4778	1.4838	440

^{*}Lattice parameter a = 8.37677 Structure—cubic

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Related Web sites

amplifier.ti.com

A web page of Sensor Scientific, Inc.:

www.sensorsci.com/letter.htm

Western Electronic Components, a U.S.-based thermistor manufacturer:

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