2014

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Recommended Citation
Subhanarayan SAHOO, S. K. S. PARASHAR, S. M. ALI. CaTiO$_3$ nano ceramic for NTCR thermistor based sensor application. Journal of Advanced Ceramics 2014, 3(2): 117-124.

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CaTiO$_3$ nano ceramic for NTCR thermistor based sensor application

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Received: November 28, 2013; Revised: February 27, 2014; Accepted: March 13, 2014  
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Abstract: It is possible to fabricate highly sensitive NTCR (negative temperature coefficient of resistance) thermistor using nano crystalline CaTiO$_3$ synthesized by high energy ball milling. Disc shaped green pellets were prepared and effects of sintering on the disc pellets were studied as thermistor by sintering the samples at 1000 °C, 1100 °C and 1200 °C. The as-prepared samples were characterized by X-ray diffraction (XRD), impedance analysis and electrical measurement. The resistivity of the prepared samples varies predictably with temperature: this makes them promising material for temperature sensor. The experimental results prove that nano crystalline CaTiO$_3$ ceramic is one kind of thermistor with exponential negative temperature coefficient of resistance in the temperature range of 300–500 °C. The samples have the advantages of rapid response, high sensitivity and capability to withstand thermal surges over the temperature range of 300–500 °C. Resistance–temperature characteristics are described by thermistor equation with thermistor constant around 4003 K to 10795 K and thermal coefficient of resistance $\alpha$ around $-1\%/\degree C$ to $-13\%/\degree C$. The activation energy is in the range of 0.34–0.93 eV. The observed thermistor parameters are found to be comparable with many of the known thermistor materials. This suggests that the electrical properties can be adjusted to desirable values by controlling the temperature parameter. The influence of fabrication process of disc thermistor and electrical properties are discussed. The study shows the potential of nano crystalline CaTiO$_3$ to act as an NTCR material for thermistor applications.

Keywords: CaTiO$_3$; thermistor; sensitivity index; temperature coefficient

1 Introduction

There is an increasing need of sensors for high temperature applications, and such components may be NTCR (negative temperature coefficient of resistance) ceramic resistors. Thermistor prepared using nano ceramics has already gained good rank in the family of advanced solid sensor technology. The name “thermistor” is derived from thermally sensitive resistance used to describe a form of resistive device that possesses a large temperature coefficient of resistance [1–4]. NTCR thermistor elements, which exhibit a decrease in electrical resistance with increasing temperature, are widely used in manufacturing temperature sensors with negative temperature coefficient. Monitoring and control of temperature are of importance in our daily life. Thermistor with NTCR has been widely used not only

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in various industrial and domestic applications pertaining to temperature monitoring, control and compensation, etc., but also in laboratory and medical procedures. NTCR thermistor is widely used in the world due to its potential use for many applications, such as temperature measurement, circuit compensation, suppression of inrush current, flow rate sensor, pressure sensor, aerospace, and automotive temperature measurement [5–7]. Along with the advances in modern technology, demand for precise temperature measurement and control has increased. Ceramic thermistor shows promising potential for such applications. Thermistor has several potential advantages over thermocouple and resistance thermometer: (i) it has higher temperature capability; (ii) since it works on resistance based measurement, it does not require special extension lead wires and conductors or cold-junction compensation; (iii) it has high sensitivity; (iv) it requires simple construction, leading to possible lower system cost [8]. NTCR thermistor is usually fabricated from a solid mixture of metallic oxides; even it is well known that most NTCR thermistor is produced from ceramic based on metal oxides with general formula ABO₃. The electrical characteristics of the NTCR ceramic theoretically can be controlled using several methods. One of the methods is heat treatment. The heat treatment theoretically changes the electrical characteristics such as electrical resistivity and thermistor constants, because the charge carrier of ceramic is dependent on the surrounding environment.

Ceramics are very useful crystalline materials because of their structural strength, thermal stability, light weight, and, for many materials, excellent electrical properties. CaTiO₃ based semiconducting ceramic has been studied as NTCR thermistor due to its interesting electrical properties. Low cost, ease of manufacturing and interesting thermistor properties make CaTiO₃ one of the most important ceramics today, which exhibits a uniform exponential decrease of resistance with increasing temperature in the range of 300–500 °C, i.e., a negative temperature coefficient of resistance which makes the oxide well suited for use as an NTCR thermistor material in specific temperature range.

A large number of titanate materials, whether pure or doped, have been examined for a wide variety of technological applications. Among titanate materials, calcium titanate with its chemical formula CaTiO₃, has been extensively investigated due to its many interesting electric properties. The demands for CaTiO₃ in electronic devices include varistors, capacitors, thermally sensitive resistor elements, electrodes, etc. In the present work, CaTiO₃ ceramic materials are prepared by solid state high energy ball milling technique. The NTCR behavior of CaTiO₃ is also investigated using Steinhart–Hart platform, since no reported study has been found to utilize this mode for analysis on CaTiO₃.

Steinhart–Hart equation—the basis for much of the terminology—is used throughout the industry to describe thermistor characteristics [9]. Alexander and MacQuarrie [10] selected the Steinhart–Hart equation and presented the procedure to calculate negative temperature coefficient of thermistor in order to improve accuracy [11]. The Steinhart–Hart equation is derived from mathematical curve fitting techniques and examination of the resistance vs. temperature characteristic of thermistor devices. Steinhart–Hart equation is in general used throughout the industry to give a single equation that relates resistance and temperature of an NTCR thermistor component, and it is used by all the thermistor manufacturers. Steinhart–Hart equation is named from two oceanographers associated with Woods Hole Oceanographic Institution in Cape Cod, Massachusetts. Every NTCR thermistor with different base resistance and β value has a unique resistance vs. temperature curve. The curve is best defined as a polynomial equation. The more terms are used in the polynomial, the more accuracy is achieved. However, for practical purposes, Steinhart–Hart equation is extremely accurate for most applications. In particular, using the plot of the natural log of resistance value vs. 1/T an equation of the following form is developed:

\[
\frac{1}{T} = A_0 + A_1 \ln R + \cdots + A_N \ln(R)^N
\]  

where \( T \) is the temperature in Kelvin; \( A_0, \ldots, A_N \) are polynomial coefficients that are mathematical constants.

The order of the polynomial used to model the relationship between \( R \) and \( T \) depends on the required accuracy of the model and the non-linearity of the relationship for a particular thermistor. It is generally accepted that the use of third order polynomial gives very good correlation with measured data, and the squared term is not significant.

We are studying properties of nano crystalline CaTiO₃ for high temperature thermistor and nano
crystalline CaTiO$_3$ should be thermally stable, so we use high energy ball milling technique for preparation of nano crystalline CaTiO$_3$. The planetary ball mill consists of gyratory shaft and a cylindrical jar, and both of them rotate simultaneously in opposite direction at high rotational speed allowing the balls to move strongly and rigorously, which lead to large impact energy between the balls and the material. There are many different techniques (such as chemical method, sol–gel, high temperature solid state reaction method, etc.) used to prepare nano crystalline CaTiO$_3$. However in high energy ball milling technique, there are lots of advantages such as reproducibility, homogeneous and room temperature preparation.

There is an increasing need for sensors for high temperature application. The current effort is directed toward making improvements in the performance and cost of high temperature application based disc NTCR thermistor. It also aims to provide generally useful information on factors that contribute to the performance and reliability. To do so, an understanding of the thermistor behavior is necessary.

The term “thermistor” refers to semiconducting ceramic materials, generally oxides, acting as sensing elements of devices for measuring temperature. Even today, extensive research on thermistor materials and their applications in different instrumentation systems are being carried out all over the world. Out of a wide range of materials investigated so far, metal oxide semiconductors have occupied a prominent position. Even though CaTiO$_3$ has been considered by a few investigators, the focus of research is very limited and the results are very far from any conclusions in respect of electrics and from the point of view of thermistor based sensor materials and their applications. The two major streams of research and development are being pursued nowadays. The first aspect is to prepare nanosize material for modern electronic applications, and the other is replacing traditional compositions that are based on toxic elements. Considering the importance of the above noted issues, we have initiated the development of lead free thermistor [12,13]. CaTiO$_3$ is low cost, and if we use this material in devices, the device cost will be decreased. It is also environment friendly, so it has more application in medical applications. If we will use this material in devices, environmental pollution will become less.

The present study focuses on the synthesis of nano crystalline CaTiO$_3$ NTCR thermistor. The experiment was done by heating green pellets made from nano crystalline CaTiO$_3$ at 1000 °C, 1100 °C and 1200 °C in air for 2 h. In this work, we investigated the performance of the thermistor in more detail. The results are very promising: it is possible to obtain good temperature sensitivity and good reproducibility of characteristics. The electrical characteristics such as resistivity, sensitivity index and temperature coefficient of the nano crystalline ceramic were studied after the fabrication process. We hope that this work is interesting from the stand point of preparing novel thermistor.

2 Experimental

In this work, starting powders of calcium oxide (CaO) and titanium dioxide (TiO$_2$) were stoichiometrically weighed to attain the molar ratio of CaO:TiO$_2$ = 1:1. Mechanical activation of the starting mixture was performed by grinding in a high energy ball mill (PM400, RETSCH) in a continual regime in air using the BPR (ball to powder ratio) of 20:1. The milling was performed with rotational speed of 300 rpm and milling time was set to 15 h with regular interval of 30 min. X-ray diffraction (XRD) analysis was performed on Philips X-ray diffractometer. The XRD patterns were recorded in the 2θ range.

Disc type thermistors were made by uniaxially compressing blend nano CaTiO$_3$ powders with a binder (2 wt% poly vinyl alcohol) in a die to disk, applying 4 t loads for 180 s by using KBr press. The green pellets directly placed on an alumina crucible were put in ASCO furnace and heated at 1000 °C, 1100 °C and 1200 °C for 2 h. We attempted to obtain ceramic showing NTCR characteristic by sintering in air. The electrodes were formed on polished ceramic surfaces for electrical measurements using a fired Ag paste at 700 °C for 15 min. The surfaces of sintered ceramic pellets were coated to form none ohmic contact. The electrical properties were determined by direct measurement of the current flowing under the impressed field of 0.1 V/mm using LCR meter (HIOKI 3532-50 LCR HiTester) from 300 °C to 500 °C.

3 Results and discussion

3.1 XRD analysis

Figure 1 shows the XRD pattern of CaTiO$_3$ ball milled for 15 h. All diffraction peaks can be assigned to the
orthorhombic structure. The XRD figure shows a single phase material with orthorhombic structure in accordance with the ICDD No. 89-56. The average crystallite size is estimated by Scherrer’s equation using the full width at half maximum (FWHM) of the most intense peak and found to be 90 nm. The lattice parameters are calculated to be $a = 5.3914(0)$ Å, $b = 5.4425(4)$ Å and $c = 7.6526(4)$ Å. The good arrangement between the experimental lattice parameter values with the respective ICDD card shows that, by high energy ball milling we are getting single phase nano CaTiO$_3$. As the reported literature, the Scherrer’s equation is described as follows:

$$D = \frac{0.9 \lambda}{B \cos \theta}$$  \hspace{1cm} (2)

where $D$ is the average crystallite size or particle size; $\lambda$ is the X-ray wavelength (0.1540593 nm); $\theta$ is the Bragg angle; and $B$ is the FWHM.

3.2 Electrical characteristics

Resistance data were found out by impedance spectroscopy method from the impedance data measured by LCR meter. Figures 2(a), 2(b) and 2(c) show Cole–Cole plots of samples sintered at different temperatures 1000 °C, 1100 °C and 1200 °C, respectively. Comparing the three graphs, the effect of sintering temperature on electrical conductivity comes into picture. All the Cole–Cole plots show similar semicircular nature. After analyzing these graphs by ZVIEW software, conclusion comes that all graphs have the equivalent circuits of single resistance parallel constant phase element (CPE). By finding these circuit parameters, we get the resistance data of all prepared samples in the specified temperature range. Conductivity becomes higher as the sintering temperature decreases. Figure 2(d) shows the

![Fig. 1 XRD pattern of nano crystalline CaTiO$_3$.](image)

![Fig. 2 (a), (b) and (c) Temperature dependant impedance plots of nano CaTiO$_3$ sintered at 1000 °C, 1100 °C and 1200 °C, respectively; (d) resistance vs. temperature graph of nano CaTiO$_3$ sintered at different temperatures.](image)
resistance vs. temperature graph in the range of 300–500 °C of samples sintered at different temperatures. The experimental data show NTCR property. The dependence of resistance on temperature approximately follows an exponential [11–18]. Hence the experimental data match with the Steinhart–Hart equation, revealing the significance of nano crystalline CaTiO3 as a promising environment friendly material for high temperature thermistor application. The experimental resistance data were got by impedance spectroscopy, and then the three point method of Steinhart–Hart equation was applied on those data. It is found that the experimental data reveal the applicability of nano crystalline CaTiO3 as NTCR. The resistance of prepared samples varies predictably with temperature: this makes them suitable for use as temperature sensor [1].

Figure 3 shows the temperature dependence of resistance of nano crystalline CaTiO3 heat treated at different temperatures. The relation between the logarithm of electrical resistivity against reciprocal of temperature shows linearity. The resistivity decreases monotonically with rising temperature, indicating that all the samples are good NTCR thermistor. This graph shows the relationship between natural logarithm of resistivity and reciprocal temperature 1/T. The plots of all the samples can be described by Arrhenius relationship [19–22]:

$$\rho = \rho_0 \exp\left(\frac{E_a}{k_B T}\right)$$

where $\rho_0$ is the resistivity at infinite temperature; $E_a$ is the electronic activation energy; and $k_B$ is the Boltzmann constant.

All the three samples exhibit different slopes, and different activation energies are observed, which are given in Table 1. The values of activation energy were calculated by the following formula:

$$E_a = k_B \times \beta$$

where $E_a$ is the activation energy; $k_B$ is the Boltzmann constant; and $\beta$ is the thermistor constant.

These observed values of activation energy in CaTiO3 nano ceramic may be because of presence of charge carrier inside the grain and some extrinsic charge carrier created due to the use of silver electrode at elevated temperature. Activation energy and hence thermistor constant show transition from higher value to lower value. Thermistor constant and activation energy are important from the point of view of applications.

As discussed before, third order polynomial gives a very good correlation with measured data, and the squared term is not significant. The Steinhart–Hart equation [23] with the squared term eliminated is the most common form of the equation used and is usually found explicit in temperature $T$. The equation is then reduced to a simpler form, and it is generally written as

$$\frac{1}{T} = a + b \ln R + c (\ln R)^3$$

where $T$ is in Kelvin unit; $a$, $b$ and $c$ are the curve fitting coefficients; and $\ln R$ is the natural logarithm of a resistance in Ohm.

The equation is relevant for the complete useful temperature range of a thermistor. The coefficients $a$, $b$ and $c$ are constants for individual thermistor. Unlike $\alpha$ and $\beta$, they should not be regarded as material constants. The equation is considered for three temperature points in the range—usually at the low end, the middle and the high end of the range. This ensures best fit along the full range. Precisely controlled measurement of temperature and associated resistance value of the thermistor is made in a temperature controlled medium at these three

### Table 1 Temperature dependant activation energy for different sintered samples

| Temperature (°C) | Sintering temperature (°C) | $\beta$ (K) $E_a$ (eV) | $\beta$ (K) $E_a$ (eV) | $\beta$ (K) $E_a$ (eV) |
|-----------------|---------------------------|------------------------|------------------------|------------------------|
|                 | 1200 °C                   | 1100 °C                | 1000 °C                |
| 300             | 5958                      | 0.51                   | 7311                   | 0.63                   | 10795                  | 0.93                   |
| 325             | 5641                      | 0.49                   | 6797                   | 0.59                   | 10319                  | 0.89                   |
| 350             | 5349                      | 0.46                   | 6320                   | 0.54                   | 9885                   | 0.85                   |
| 375             | 5081                      | 0.44                   | 5875                   | 0.51                   | 9490                   | 0.82                   |
| 400             | 4833                      | 0.42                   | 5458                   | 0.47                   | 9128                   | 0.79                   |
| 425             | 4603                      | 0.40                   | 5065                   | 0.44                   | 8797                   | 0.76                   |
| 450             | 4390                      | 0.38                   | 4894                   | 0.40                   | 8493                   | 0.73                   |
| 475             | 4191                      | 0.36                   | 4341                   | 0.37                   | 8213                   | 0.71                   |
| 500             | 4006                      | 0.35                   | 4003                   | 0.34                   | 7955                   | 0.69                   |
calibration points. It should be noted that Steinhart–Hart equation produces a good approximation to the relation between \( T \) and \( R \) for the complete range of a thermistor based on data from just three calibration points. Solving this equation set, we obtain values for fitting coefficients of Steinhart–Hart equation shown in Table 2. Table 2 shows how the fitting coefficients of Steinhart–Hart equation are varied for the same nano crystalline CaTiO\(_3\) only because of the variation of thermal treatment.

The nano crystalline CaTiO\(_3\) ceramic has been preferred for thermistor due to gentle slope of \( \beta \) factor. \( \beta \) value is a very important parameter in description and specification of thermistor materials and thermistor components. It is the exponential factor also known as sensitivity index of thermistor materials. Figure 4 shows \( \beta \) value in the temperature range of 300–500 \( ^\circ \)C of sintered samples at different temperatures. If \( \ln R \) is plotted versus \( 1/T \), then the slope of the resulting graph will be equal to \( \beta \). \( \beta \) value can be used to calculate resistance or temperature value, and regarded as a quantitative value of thermistor materials that is assigned as a material constant indicating the relationship of material resistance to temperature. \( \beta \) value is derived from a mathematical approximation. For this mathematical approximation to apply over a large temperature range, \( \beta \) value has to vary with temperature. The variation of \( \beta \) value with temperature is indicated in the graph shown in Fig. 4. \( \beta \) value is also used to calculate \( \alpha \) value (temperature coefficient) for a thermistor made from the same material. \( \beta \) value is a single expression that can be regarded as a material constant. It depends on basic material properties, and \( \beta \) value derived from measurements provides an indication of general thermistor material quality. The equation for \( \beta \) value using Steinhart–Hart coefficients is

\[
\beta = \left( \frac{b}{3c} \right)^{\frac{1}{3}} + \frac{\alpha^2}{4}
\]

(6)

The sensitivity of a thermistor for temperature sensing is described by the activation energy of electrical conduction, as obtained from negative slope of the Arrhenius plot of logarithm of electrical resistivity vs. reciprocal of absolute temperature. The activation energy reflects the energy for the hopping of charge carrier in the prepared samples [24].

\( \alpha \), a material characteristic, shows the percentage resistance change per degree centigrade [25, 26]. It is also referred as the temperature coefficient. The temperature coefficient is a basic concept in thermistor calculation. Because the resistance of NTCR thermistor is a function of temperature, the \( \alpha \) value of a particular thermistor material is also nonlinear across the relevant temperature range. The above statement is true if Fig. 2(d) and Fig. 5 are compared. It shows all the three samples showing rapid thermal response. The equation for \( \alpha \) value using Steinhart–Hart coefficients is

\[
\alpha = \frac{a - 1}{T} = -\frac{\beta}{T} \times 100
\]

(7)

For the usual NTCR thermistor materials, \( \beta \) constant is closely related to resistivity as shown in Fig. 6, so the search for different pairs requires complete formulation changes. \( \beta \) value is a quantitative value

### Table 2 Temperature dependant Steinhart–Hart coefficients for different sintered samples

| Sintering temperature (\(^\circ\)C) | \( a \) | \( b \) | \( c \) |
|-----------------------------------|--------|--------|--------|
| 1200                              | 0.000416221 | 0.000034033 | 0.000000113 |
| 1100                              | 0.000283495 | 0.000066091 | 0.000000057 |
| 1000                              | 0.000399745 | 0.000057168 | 0.000000069 |

Fig. 4 Typical temperature dependence of \( \beta \) constant.

Fig. 5 Typical temperature coefficient of resistance.
of thermistor materials that is assigned as a material constant and indicates the relationship of material resistivity to temperature. The general information on sensitivity of material resistivity to temperature that can be interpreted from \( \beta \) value is indicated in Fig. 6.

\[
R = A \exp \left( \frac{\beta}{T} \right) \tag{8}
\]

where \( R \) is the resistance; \( T \) is the temperature; and \( \beta \) is the exponential factor known as sensitivity index of the thermistor material.

\( \beta \) value is a very important parameter in the description and specification of thermistor materials and thermistor components. When the natural logarithm of both sides of Eq. (8) is taken, the relationship becomes

\[
\ln R = C + \frac{\beta}{T} \tag{9}
\]

where \( C \) is a constant factor, \( C = \ln A \) from the equation above.

The above equation shows the linear relation between \( \beta \) value and natural logarithm of resistivity, which we can realize from Fig. 6. Although this simple model for the relationship between resistance and temperature of a thermistor is limited over large temperature spans, concepts derived from it are of importance in thermistor industry and in the specification of NTCR thermistor. Some of these concepts are developed with the intention of explaining some of the basic calculations and specifications used in the industry.

4 Conclusions

This paper provides a new class of thermistor. The potential of nano ceramic CaTiO\(_3\) as thermistor based sensor material has been investigated. It is possible to obtain high temperature sensitivity using this material. Since temperature is one of the most important factors, temperature sensors have been used in many applications. In particular, a high performance NTCR thermistor is important because the thermistor should detect temperature over a wide range and under very severe condition. Resistance–temperature characteristics can be described by thermistor equation Steinhart–Hart model with thermistor constant \( \beta \) around 4000 K to 15000 K, which is good for thermal sensing applications. As we know, if a material has high \( \beta \) value, it has high temperature stability property. Here we get high \( \beta \) value in the range of 4003 K to 10795 K, so it means our material is capable to withstand temperature surges and very suitable in high temperature electronic applications. Comparisons of properties are made between NTCR thermistors consisting of nano ceramic CaTiO\(_3\) with different heat treatment. All three thermistors are obeying Steinhart–Hart model, which means all the three samples are useful for high temperature thermistor based sensor applications with rapid thermal response. The characteristics of thermistor for temperature measurement are the good sensitivity to yield high resolution, high accuracy, and reproducibility in the specified temperature range.

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