Investigation of electrical characteristics of different ceramic samples using Hall effect measurement

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Abstract

Hall effect is a very popular technique and is widely used to quantify important electrical parameters such as carrier concentration, resistivity, mobility and Hall coefficient of different types of samples. In this paper, electrical characteristics of three ceramic samples will be analyzed using a Hall effect measurement system (Ecopia, HMS-3000), which is based on the van der Pauw method. Measured results for mobility and electrical resistivity at three temperatures (25°C, 50°C and 80°C) will be presented. Current-voltage and current-resistance dependence between terminals of four point contact of different samples will be also demonstrated.

Keywords: nickel-manganates, Hall effect measurement

I. Introduction

The electrical characterization of materials have evolved from the resistance *R* and the conductance *G* measurement to an intrinsic material property like resistivity (or conductivity), because the resistance alone was not comprehensive enough since different sample shapes can give different resistance values. Since different materials (semiconductors) can have the same resistivity, and also for a given material, different values of resistivity can be found, depending on how it is processed, then resistivity is not a fundamental material parameter. In order to determine both the mobility and the sheet concentration of some material, a combination of a resistivity measurement and a Hall measurement is needed. Because of its simplicity, low cost, and fast turnaround time, the Hall effect measurement is an indispensable characterization technique in the semiconductor industry and in research laboratories.

Hall measurement was used in material investigation often, for semiconductors especially [1]. It has been used for investigation of polycrystalline and superconductivity [2,3], canonical spin glass [4], superconductors based on semiconductor (Si, Ge) substrate [5], and high temperature superconductors [2]. Lately, the quantum Hall effect [6,7] has become very interesting.

In this paper, we have measured the electrical characteristics of ceramic samples with the composition of $NiMn_2O_4$ but with different sintering temperature and time. The bulk and sheet carrier concentration, mobility, resistivity and average Hall coefficient of samples have been measured and compared at three different temperatures 25°C, 50°C and 80°C. The standard van der Pauw technique with four omic contact has been used to determine the resistivity of uniform samples. Hall measurement is based on Hall effect phenomena, which is shortly described in the next section.

II. Theoretical Background

Van der Pauw method

The van der Pauw method is a commonly used technique, based around the Hall effect, which characterises a sample of semiconductor or similar material. The van der Pauw measurement technique can be used for the determination of the resistivity on a sample of arbitrary shape by using four small contacts placed on the periphery, as shown in Fig. 1. Additionally, the following material parameters can be determined by means of this technique:

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the sheet resistance; the doping type (i.e. if it is a *p*-type or *n*-type) material; the sheet carrier concentration of the majority carrier (the number of majority carriers per unit area, the doping level, can be found for a sample with a given thickness); the mobility of the majority carrier.

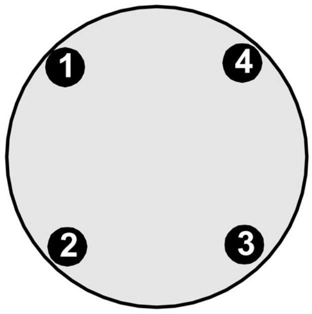

Figure 1. Labelled contacts of circular shaped sample

For the Hall effect experiment, the contacts are usually numbered from 1 to 4 in a counter-clockwise order, beginning at the top-left contact as it is depicted in Fig. 1 for a circular shaped sample. The current I_{12} is a positive DC current injected into contact 1 and taken out of contact 2. The voltage V_{34} is a DC voltage measured between contacts 3 and 4 with no externally applied magnetic field.

Resistivity measurements

The objective of the resistivity measurement is to determine the sheet resistance R_S. To make a measurement, a current caused to flow along one edge of the sample (for instance, I_{12}) and the voltage across the opposite edge (in this case, V_{34}) are measured. From these two values, a resistance (for this example, $R_{12,34}$) can be found using Ohm's law: $R_{12,34}=V_{34}/I_{12}$. The sheet resistance of samples with arbitrary shape can be determined from two of these resistances - one measured along a vertical edge, such as $R_{12,34}$, and a corresponding one measured along a horizontal edge, such as $R_{23,41}$. The actual sheet resistance is related to these resistances by the van der Pauw formula.

A further improvement in the accuracy of the resistance values can be obtained by reciprocal measurements ($R_{AB,CD} = R_{CD,AB}$) and reversed polarity measurements.

$$R_{vertical} = \frac{R_{12,34} + R_{34,12} + R_{21,43} + R_{43,21}}{4}$$

and (1)

$$R_{horizontal} = \frac{R_{23,41} + R_{41,23} + R_{32,14} + R_{14,32}}{4}$$

Van der Pauw formula becomes:

$$e^{-\pi R_{vertical}/R_S} + e^{-\pi R_{horizontal}/R_S} = 1 \tag{2}$$

An iterative method (nested intervals) is used to solve the van der Pauw formula numerically for sheet resistance R_S.

Hall measurements

The objective of the Hall measurement in the van der Pauw technique is to determine the sheet carrier concentration n_s by measuring the Hall voltage V_H. The Hall voltage can be defined in terms of the sheet concentration:

$$V_H = \frac{IB}{qn_S}$$

In this paper, Hall voltage was measured by a Hall Effect Measurement System, HMS-3000. A formula for the majority carrier mobility can be given in terms of the previously calculated and measured sheet resistance and sheet concentration:

$$\mu_m = \frac{1}{qn_S R_S}$$

If the sample thickness *d* is known, the bulk resistivity ($\rho = R_S d$) and the bulk concentration ($N_B = n_S/d$) can be determined.

III. Measuring Technique

Measuring system used in this paper, depicted in Fig. 2, has a 0.37 Tesla permanent magnet. Hall measurements can be performed by altering the incoming current until the voltage produced is near some target voltage. In order to obtain the target voltages for a highly resistive sample, the current applied must be very small.

The measurements require four ohmic contacts to be placed on the sample. In addition to this, any leads from the contacts should be constructed from the same batch of wire to minimise thermoelectric effects. For the same reason, all four contacts should be of the same material. Contacts were provided by copper wires soldered with indium/tin pellets to the corners of the sample. The size of the contact spots was made as small as possible, less then 0.5 mm, to increase the accuracy of measurements that drops as the ratio of the contact area to the sample area increases.

In order to use the van der Pauw method, the sample thickness must be much less than the width and length of the sample. It is preferable to fabricate samples from thin plates of the ceramic material and to adopt a suitable geometry. In order to reduce errors in the calculations, it is desirable that the sample is symmetrical. There must also be no isolated holes within the sample. It is possible to test various samples using two kinds of sample board (20 mm × 20 mm or 6 mm × 6 mm), as can be seen in Fig. 3. Samples need to be soldered in four points edge.

All ceramic samples have the same composition $NiMn_2O_4$, but they have different sintering temperature and sintering time.

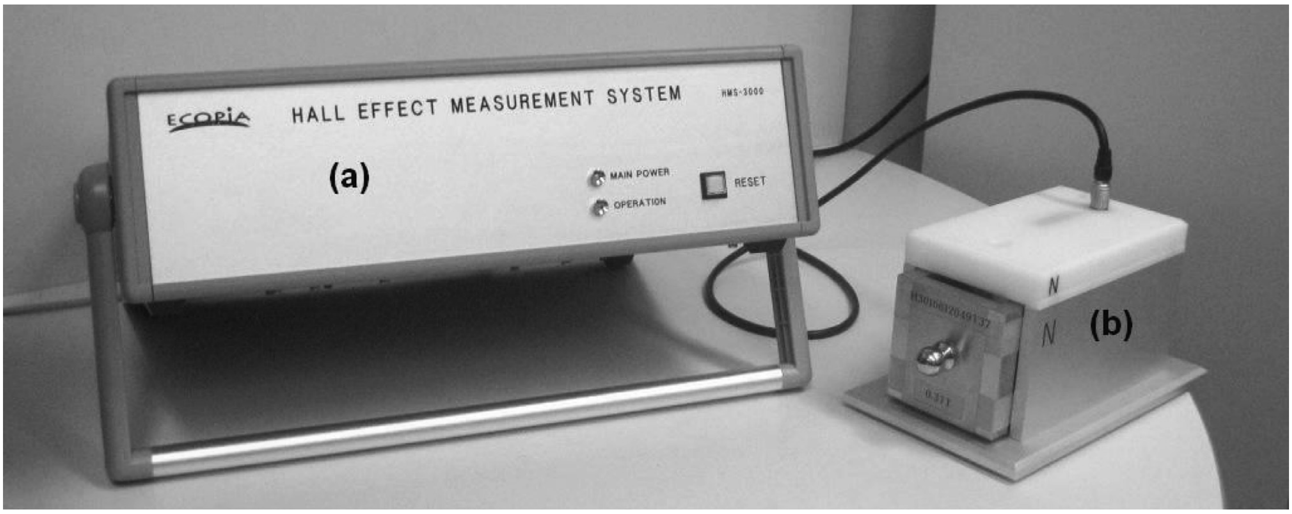

Figure 2. Hall effect measurement system HMS-3000

NTC thermistor powder was prepared of a Mn, Ni and Co–oxides mixture using a classical powder preparing procedure such as calcinating and ball milling. The composition comprising of Mn : Ni oxides in the ratio 4 : 1 and a small amount of Fe and CoO oxides was calcinated at 1050°C for 1 hour. After vibratory, ball and ultra fast milling an average powder particle size of 0.9 μm was achieved. Disc shaped pellets of the powder were pressed at a pressure of 2.5 MPa. The green samples were sintered in the temperature range of 1200–1400°C during 30–120 min. Afterwards the samples were polished.

In this paper, three samples have been chosen and they are labelled as follow:

- Sample 1: $NiMn_2O_4$, sintering time/temperature: 30 min/1200°C, thickness: 1.34 mm;
- Sample 2: $NiMn_2O_4$, sintering time/temperature: 30 min/1400°C, thickness: 1.56 mm;
- Sample 3: $NiMn_2O_4$, sintering time/temperature: 120 min/1300°C, thickness: 1.42 mm

IV. Results and Discussion

The measurements of all samples have been performed at three different temperatures: room temperature, 50°C and 80°C. The sample board (holder) needs to be inserted into magnet set lid, which is shown in Fig. 2(b). After closing the lid of magnet set, sample is heated to requested temperature.

The complete measurement process is controlled by HMS-3000 software tool. The main screen of this software tool contains input value, measurement data and result sections as well as command button panel. In the input value section, data for applied magnetic flux density, input current, temperature and sample thickness can be entered. In the result section, measured data can be seen such as bulk and sheet carrier concentration, resistivity, conductivity, mobility, average Hall coefficient, *A-C* and *B-D* cross Hall coefficient, alpha (vertical/horizontal ration of resistance) or magneto-resistance.

In addition to this, the user-friendly software tool for HMS-3000 has a possibility of giving the graphical illustration of the measured values in the form of *I-V* curve and *I-R* curve. The curves on *I-V* graph are approximately linear, as it is expected. The obtained resistance, for all analyzed samples, on *I-R* graph is almost constant if applied current is in the range from -50 nA to 50 nA (in this case), and it is around 10 MΩ.

Measured results of the mobility as a function of temperature for all three samples are presented in Fig. 4. It is well known that $NiMn_2O_4$ samples are polycrystalline. Polycrystalline samples have trap majority carriers at the grain boundary from the adjacent grain. This creates potential barriers across grain boundaries, which affects majority carrier mobility. The increase in temperature decreases the height of potential barriers leading to increase in mobility with temperature. Therefore,

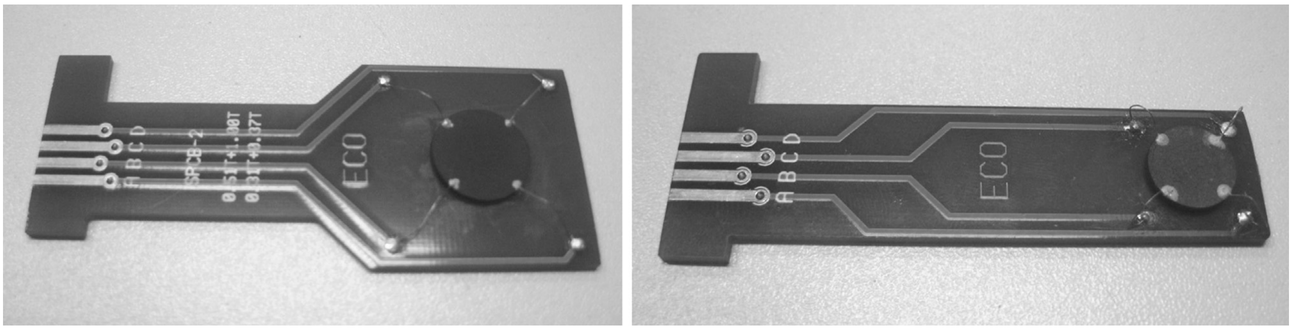

Figure 3. Analyzed samples on the two types of PCB holders

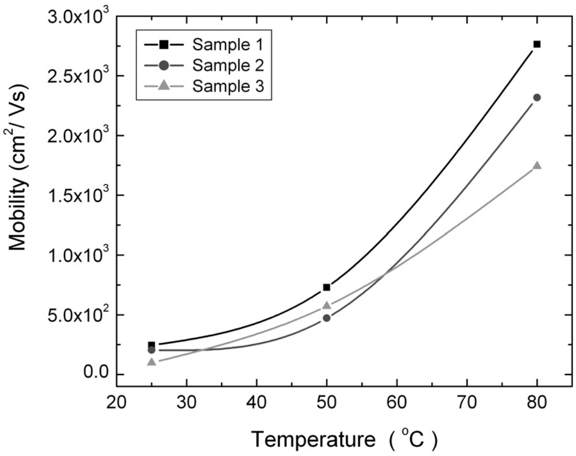

Figure 4. The mobility (μ) as a function of temperature for three analyzed samples

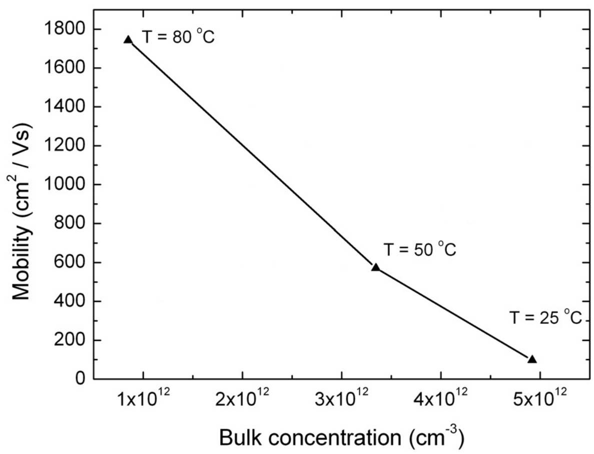

Figure 6. Mobility as a function of the bulk concentration varied with temperature for sample 3

it can be seen from Fig. 4 that mobility of each sample increases with an increase of temperature and the sample 1 (sintered at the lowest temperature) has the highest value of the mobility.

The variation of resistivity (ρ) for each sample with temperature is shown in Fig. 5. It is well-known that thermistors with negative temperature coefficient (NTC) are semiconducting oxides whose composition is a mixture of transition metal oxides (manganese, cobalt, copper and nickel) [8–10]. The primary characteristic of these electroceramics is their capability for displaying substantial change in electrical resistance with a change in temperature. Our results show that analyzed ceramic samples (powders) exhibit a decrease in electrical resistivity value as the temperature (T) increases. This means that ceramic material presented in this paper can be used for manufacturing NTC thermistors. All three samples have linear relationship between log ρ and $1/T$, but the samples 2 and 3 demonstrate a greater variation of resistivity with the temperature change.

The mobility as a function of the bulk concentration is shown in Fig. 6, for the sample 3 and three different temperatures. When the concentration increases, the mobility is decreased by the effect of ionization scattering. From Fig. 6 it can be noticed that bulk concentration of the sample 3 is around 10^{12} cm^{-3} and similar values have been obtained for the other analyzed samples.

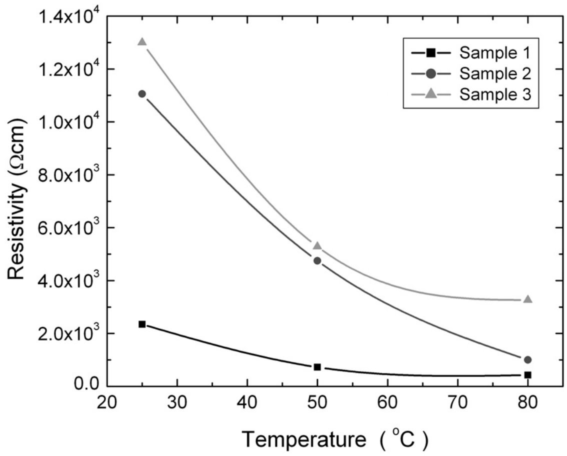

Figure 5. Relationship between the electrical resistivity (ρ) and temperature for three samples

V. Conclusions

The important electrical parameters such as mobility, resistivity and bulk concentration for $NiMn_2O_4$ ceramic samples with different sintering temperature and time were analyzed using the Hall effect measurement system. The measurement shows that different temperatures of samples are influenced on results (N_B, n_S, μ, ρ and R_H) for each sample. The presented ceramic samples have demonstrated a negative temperature coefficient and they can be used for the fabrication of NTC thermistors. Results also approved that HMS-3000 is able to measure characteristics of ceramic materials successfully if the resistance of a sample is not over 20 MΩ.

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