Effect of The Nickel And Temperature Dependent Electrical Properties C-SiO₂ -Ni Composites

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Abstract

The sol-gel method was chosen to synthesize C-SiO₂/Ni nanocomposites, silica nanofillers were incorporated into a carbon based on resorcinol-formaldehyde (RF), doped with 5% nickel. During preparation process, they were subjected to a heat treatment of different pyrolysis temperatures and under an inert atmosphere for 2 h. The X-ray diffractogram presented by *XRD* of the samples treated at low temperatures, indicates the presence of characteristic lines of metallic nickel. FTIR analysis shows the presence of a main band located at about 1050 cm⁻¹, which corresponds to the vibrations of Si-O-Si. From electrical characterizations, the C-SiO₂-Ni_{5%}-650 sample has a negative differential resistance behavior (NDR) at low measurement temperatures. According to the I-V characterization, the C-SiO₂-Ni_{5%}-625 °C nanocomposite reveals the NDR behavior at room temperature. The conduction mechanism was fitted by two models: the hopping conduction model for the nanocomposite, treated at 650 °C, and the small polaron model for the composite treated at 675 °C.

Introduction

Studying the negative differential resistance (*NDR*) behavior as well as its origins has been attracting much interest thanks to its importance in many applications of nano-electronics. The NDR behavior was originally observed in quantum mechanics, which have the possibility of obtaining it in nano-electronic devices. To date, such behavior has been observed and predicted in molecular electronic devices [1–6], and in functionalized graphene nano-ribbons [7–10] and in carbon nanotubes (*CNTs*) [11]. Researchers are interested in nanoscale devices because they have high packaging density and are more valid than microelectronic devices. For different electronic components, the NDR behavior becomes the most important property of electronic transport [12]. The NDR behavior has attracted the attention of scientific community because of its wide applications range. That is to say, NDR devices are multifunctional. They are used in several fields, such as, electronics, amplification, oscillation, switching and electronic devices [13–15]. Nowadays, many researchers have been reported on the origin and on the application of the negative differential resistance behavior. Of them, Ben Mansour et al. [16] have observed the NDR behavior in PF/ NiO nanocomposites, produced by the sol-gel process the existence of an NDR at 300 K. They have explained by the electro-thermal model. A study by Sang et al. [17] have shown that a n-ZnO nanorods (NRs) / p-degenerate diamond tunnel diode exhibits NDR behavior, properties which depends on temperature and tunnel injection behaviors carrier. At high biases, Venkataraman et al. [18] have proven nonlinear NDR and hysteresis behaviors in self-assembled benzenedithiol-gold nanoparticles. Such behaviors can be assigned to a combination of field assisted tunneling and charge trapping in nanoscale arrays. Directed self-assembly of molecular electronic arrays of benzenedithiol metal nanoparticles is suggested for molecular integrated circuits in applications such as memory, switching, hardware security, and computing. Luican-Mayer et al. [19] have observed that the NDR behavior has been observed in the transition metal dichalcogenide material. They have ascribed it to the interaction of inter-layer and intra-layer tunnels with the participation of atomically localized states of charge density wave maxima and minima. Such coupling can be also affected by network faults. Khan et al. [20] have

confirmed the presence of NDR behavior in metal doped semiconductor materials based NiO powder. According to them, this behavior is due to tunnel current.

In this work, we present a conductive polymer composite composed of a conductive carbon polymer matrix based on the resorcinol-formaldehyde (RF) precursor, incorporated by 30% of silica nano-filler reinforced with 5% of nickel elaborated by the sol-gel process. The effect of the addition of SiO₂ nanoparticles was well studied by Gouadria et al. [21]. They have stated that samples, treated at low pyrolysis temperatures, possess the NDR behavior at low measurement temperatures. Their results also indicated that the successful incorporation of nano-SiO₂ particles in carbon composite by sol-gel method improves the performance of the latter. In this work, we aim to study the effect of nickel on the electrical properties of samples treated at low pyrolysis temperatures, which exhibit an NDR behavior at ambient temperature [21, 22]. In addition, morphological and structural and electrical characterizations were conducted on our samples. The NDR at room temperature was discussed.

Experimental Work

Previous work by Gouadria et al. [21] synthesized non-doped nanocomposites based on an organic carbon matrix from Resorcinol-Formaldehyde (RF) incorporated by inorganic reinforcement such as silica (SiO₂) [21, 22]. In the present work, C-SiO₂-Ni_{5%} nanocomposites were prepared by the sol-gel method in three steps. The first step consists in developing the SiO₂ nanoparticles by the sol gel method. We dissolve the TEOS (*Tetra-Ethyl-Ortho-Silicate*) Si (C₂H₅O)₄ was dissolved in methanol with magnetic agitation at room temperature for 15 minutes, supercritical drying was applied by putting the obtained mixture in an autoclave at a precise temperature and pressure ($T_c = 250^{\circ}\text{C}$, $P_c = 7 \text{ MPa}$) in order to extract the solvent. The second step consists in synthesizing the organic carbon matrix based on resorcinol formaldehyde (RF). The latter was prepared by mixing 12.5 g of resorcinol (C₆H₄(OH)₂, 99%, *Fisher Scientific*) are mixed with 22.7 mL of formaldehyde (CH₂O, 99% *Bio pharm*) then a picric acid catalyst (C₆H₃N₃O₇, 99% *Scharlau*) with magnetic mixer for 15 min. The solution of the silica reinforcements, obtained in the first step, and the carbon matrix, obtained in the second step, were mixed to obtain the desired nanocomposite then these nanocomposites are doped with 5% of the nickel (*of the total mass of the solution*). This mixture was placed under magnetic stirring for one hour. In order to obtain the gel, the obtained solution was poured into tubes and treated in a water bath at a temperature of 50°C. By removing water and diluted alcohol, a xerogel is obtained. The obtained samples have an organic monolith form. To maintain such form, they were placed in a humid atmosphere at 50°C for a few days. Then a heat treatment is applied to these samples at 50°C for 1 day, by increasing the temperature 10°C every day until 150°C to extract the solvent from the pores. Finally, the samples were subjected to a heat treatment from 600 to 1400°C. The doped C-SiO₂-Ni_{5%} nanocomposites have a monolith form.

The synthesized C-SiO₂-Ni_{5%} nanocomposites were characterized by X-ray diffraction (*D8-Advance, Bruker diffractometer*). The morphology of the C-SiO₂-Ni_{5%} nanocomposites was studied by transmission electron microscopy (TEM-*JEOL 2100, FEG-TEM 200 kV*) and from scanning electron microscopy (SEM,

JEOL JSM-5800 LV). The Fourier transform infrared spectroscopic (FTIR) measurements were performed over the frequency range ($400-4000\text{ cm}^{-1}$) using a Vertex 70 (Bruker) spectrometer.

Electrical measurements are carried out under vacuum using a liquid nitrogen cryostat for measurement temperatures in a range of 80 to 300 K. V-I measurements were made using a computer-linked computer system comprising an Agilent 34401A multimeter and a Keithley 220 current source. Using a frequency range of 40 Hz to 100 MHz, the AC impedance measurements were performed using an Agilent 4294A impedance analyzer. To measure the conductance G in parallel mode, an AC signal is used with a voltage amplitude of 50 mV.

Results And Discussion

3.1. XRD analysis

X-ray diffraction patterns of C-SiO₂-Ni_{5%}-625, C-SiO₂-Ni_{5%}-650 and C-SiO₂-Ni_{5%}-675 nanocomposites are illustrated in Fig. 1. Previous studies by Gouadria et al. [22] indicated that the composites are amorphous at different temperatures. However, the idea of the incorporation of the nickel is to improve the conductivity of the materials. Min Zhang et al. [23] have studied the synthesis of SiO₂/C-PdNi composite. They have observed Ni particles on the surface of a sample with a size of 290 nm. On the other hand, this spectrum that Ni has improved the crystalline structure of these samples, the characteristic peaks were detected at $2\theta = 44.5^\circ$, 51.9° and 76.5° which may correspond to crystalline planes of metallic Ni phases (JCPDS 01-1260) [24, 25], respectively at (111), (200) and (222). The appearance of two large peaks corresponding to the silica structure at approximately 21.7° (*phase amorphous silica*) and 12° (*phase carbon*) [26].

3.2. TEM micrograph

Microscopic electronic transmission (TEM) characterizations are performed on nanocomposites treated at different pyrolysis temperatures. TEM photograph is shown in Fig. 2. The samples (Fig. 2.a, b, c) show homogeneous interconnected nano-sized spherical particles in the range of 23–30 nm. The TEM micrograph of the sample processed at 675 exhibits the existence of a percolation phenomenon (Fig. 2.c). High resolution TEM images are presented at the right of each image. We can clearly see in these TEM images that the nanoparticles agglomerate by increasing the pyrolysis temperature, this agglomeration can be explained by the interaction between the matrix and the nanoparticle. Lei Ding et al. [27] studied the effects of the molar ratio of resorcinol to nickel ion on the SiO₂/C-Ni composite and they found that this material showed a because of the good dispersion of Ni particle, the sample display excellent performance for protein adsorption.

Figure 3 shows Scanning Electron Microscopy (SEM) images and Energy-Dispersive X-ray (EDX) graph of SiO₂/C-Ni nanocomposites samples. These analyses confirmed that the elements are only Ni, C, Si and O (Fig. 3). EDX confirmed purity of SiO₂/C-Ni. Based on these results, it appears that silica particles can

surround, carbon chains and become incorporated into pores, which can lead to changes in the material's textural and electrical properties. In addition to this, you need to know more about it. Previous studies done by Gouadria et al. [28] shows the same results of this agglomeration of nanoparticles but the addition of Nickel, in this work, favors the agglomerations of nanoparticles at low pyrolysis temperatures where Nickel increased the conductivity of these materials.

3.3. FTIR analysis

The FTIR spectrum of the nanocomposite processed at the 625 °C in 400–4000 cm^{-1} range is shown in Fig. 4. Four major peaks were detected. A Ni–O bond was detected corresponding to 470 cm^{-1} , also an H–OH bond (*water molecules*) was observed at 1600 cm^{-1} [29, 30]. We notice the appearance of the Si–O–Si, Si–C bands are appearing at 801 cm^{-1} [31]. In addition, a large band at approximately 1050 cm^{-1} corresponds to the Si–O–Si or ethoxy groups attached to Si [32, 33]. Shahrokh Abadi et al. [34] studied the effects of the annealing temperature on the silica and they found that this material showed a band at 1110–1070 cm^{-1} are interpreted with the Si–O–Si asymmetric bond.

3.4. V-I characterizes

The characteristics V(I) was studied according to the pyrolysis temperature and the measuring temperature for the C-SiO₂-Ni_{5%}-625, C-SiO₂-Ni_{5%}-650 and C-SiO₂-Ni_{5%}-675 nanocomposites are shown in Fig. 5. Two different characteristics appear: an ohmic region appears at low current and an NDR region exists at high voltage, the NDR behavior appears at low measuring temperature in the samples C-SiO₂-Ni_{5%}-650 (Fig. 5.b) in the range of 80 to 160 K. Also, the NDR appears only in the range 80 K – 120 K for (C-SiO₂-Ni_{5%}-675 (Fig. 5.c)) composite. However, the NDR persists even at room temperature in the sample treated at 625 (C-SiO₂-Ni_{5%}-650 (Fig. 5.a)). Many authors have been explained the existence of the NDR phenomenon by the model electro-thermal for the samples materials [35, 36]. We notice that the existence of the NDR is correlated with the conductivity of the material, in fact, for samples with high resistivity the NDR persists at ambient temperature, is the case for C-SiO₂-Ni_{5%}-625 which has a conductivity equal to $\sigma = 1.634 \cdot 10^{-5} (\Omega \cdot \text{cm})^{-1}$ [37, 38]. While when conductivity increases the NDR does not appear at ambient temperature and only appears for low measuring temperatures, is the case for C-SiO₂-Ni_{5%}-650 ($\sigma = 1.54 \cdot 10^{-3} (\Omega \cdot \text{cm})^{-1}$ [39]) and for C-SiO₂-Ni_{5%}-675 ($\sigma = 4.1 \cdot 10^{-3} (\Omega \cdot \text{cm})^{-1}$ [38, 40]). As a conclusion of our work, we can explain that insulating materials have the advantage of the appearance of the NDR at ambient temperature. This is why we are interested in the sample processed at 625°C. Xia et al. [41] studied the SiO₂/C materials composites and they found that enhanced electrochemical performance can be associated with their porous sample, which can improve electrical conductivity of the composite. The novelty in this article is that the NDR phase exists at room temperature and at low pyrolysis temperature, this allows to use in negatronic applications. The results of the analysis of conductance ac through the frequency for diverse samples are illustrated in Fig. 6. The ac conductance in the high range frequency obeys Jonscher's law according to Eq. (1) [42]:

$$G(\omega) = G_{dc} + A\omega^s \text{ Eq. (1)}$$

where G_{dc} shows dc conductance, A is a pre-exponent factor and s is the frequency exponent. In order to know the conduction mechanism in a material, several theoretical models have been used to show the temperature dependence with the exponent s . For CBH mechanism, s decreases by increasing the temperature [43, 44]. For Small Polaron Mechanism (SP), s increases with increasing the temperature [45, 46]. Following the OLPT (*Overlapping large polaron tunnelling*) mechanism, s decreases when the temperature increases, and shows a minimum for a precise temperature and then enlarges when the temperature increases [47, 48]. In quantum mechanical tunnel effect model (*QMT*), s does not vary with temperature and almost equal to 0.8 [49–51]. Previous studies done by Gouadria et al. [39] on nanocomposite based on a carbon matrix doped with 50% silica shows the existence of a quantum mechanical tunnel model (*QMT*) for the sample treated at 675°C whose the frequency exponent is independent of the temperature and almost equal to 0,87. Previous studies by Ben Mansour et al. [16] on nanocomposite based on pyrogallol and formaldehyde (PF) based carbon matrix doped by Nickel oxide (NiO) processed at 625 indicates the presence of a small polaron hopping model.

A linear adjustment of Eq. (1), gives the values of s are illustrated in Fig. 7. For the nanocomposite C-SiO₂-Ni_{5%}-650 (Fig. 7.a), the values of s vary between 0 and 1, indicating the presence of the hopping conduction mechanism. However, for the nanocomposite C-SiO₂-Ni_{5%}-675 (Fig. 7.b), s increases with the measurement temperature. It is clear, that the SPH model of this composite material can be dominant. The addition of nickel promotes percolation sites and allows the electron to jump from site to another so the conduction pattern in the samples changes as the pyrolysis temperature varied.

Conclusion

The effect of nickel doping on the structural and physical properties of C-SiO₂-Ni_{5%} nanocomposites materials produced by the sol-gel process was studied. The incorporation of Nickel promotes the appearance of characteristic metallic Nickel lines in the XRD analysis at low pyrolysis temperature. The electrical analysis demonstrated the overcome of two conduction mechanisms as a various temperatures; a hopping conduction model in C-SiO₂-Ni_{5%}-650°C sample and the SPH model for the nanocomposite C-SiO₂-Ni_{5%}-675°C. The characteristics $V(I)$ at the ambient measuring temperature show a negative differential resistance for a low pyrolysis temperature, NDR phenomenon is correlated with the resistivity of the materials and has an electro thermal origin. In addition, the analysis of the electrical properties shows exceptional performance for these nanocomposites, considering the low cost of the method of sample elaboration. This new C-SiO₂-Ni sample is a suitable candidate in switching and electronic devices.

Declarations

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Figures

Figure 1
 XRD spectrum of C-SiO₂-Ni5%-625, C-SiO₂-Ni5%-650 and C-SiO₂-Ni5%-675 nanocomposites

Figure 2

TEM photograph of (a): C-SiO₂-Ni5%-625, (b): C-SiO₂-Ni5%-650 and (c): C-SiO₂-Ni5%-675 nanocomposites

Figure 3

SEM and EDX analysis of C-SiO₂-Ni5% nanocomposites

Figure 4

FTIR spectrum of C-SiO₂-Ni5%-625 °C nanocomposite

Figure 5

The variation of $V(I)$ analysis versus measurement temperatures for the (a): C-SiO₂-Ni5%-625, (b): C-SiO₂-Ni5%-650 and (c): C-SiO₂-Ni5%-675 nanocomposites samples.

Figure 6

The ac conductivity dependence with the diverse temperatures for the (a): C-SiO₂-Ni5%-650 and (b): C-SiO₂-Ni5%-675 nanocomposites

Figure 7

Variation of exponent s versus measurement temperature of (a): C-SiO₂-Ni5%-650 and (b): C-SiO₂-Ni5%-675 nanocomposites samples.