Journal of Interfaces, Thin films, and Low dimensional systems 2 (1) Summer and Autumn (2018) 123-126

Nano dispersed metal-ceramic composite materials of the Ni-SiO_2 system

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ARTICLE INFO

Article history:
Received 21 January 2019
Revised 27 May 2019
Accepted 29 July 2019
Available online 12 Aug 2019

Keywords:
Nanopowders
Nanocomposite
Sol-Gel Method
XRD technique

ABSTRACT

In the organic field effect transistors (OFETs) generation, the silicon gate oxide is 1-2 nm thick. A shrinking of this thickness down to less than 1 nm for the next generation will lead to a couple of orders of magnitude increase in tunneling as well as leakage currents. NiO-SiO_2 can be used in a variety of devices, such as in circuit boards and detectors, including sensors, due to its porous structure. Owing to these specific properties, these composites attract the attention of many researches. The methods of sol-gel with using XRD (X-ray Diffraction) technique are used to determine the optimum conditions of obtaining composition and conditions of metallization. The obtained results show that increase in silicon oxide content in samples up to 10 wt. % would lead to almost complete the recrystallization of nickel particles at 50 °C.

1 Introduction

Nowadays there is special interest for the formation of nano composites of Nickel nanoparticles into SiO_2 matrix [1-5] due to their many applications. To study the effect of Ni Nano particles on NiO-SiO_2 Nano composite, we have demonstrated a series of experiments to synthesize different size and structured shape of NiO-SiO_2 composition. Some researchers ([1] and references therein) believe that these compositions which are synthesized with sol-gel technology show porous structures and can absorb gases very well.

To increase the thermal stability of the structure and to suppress the weakening effects related to simultaneous influence of temperature [2], smaller particle sizes are desirable because it can show leakage current and diffusion through the ultrathin silicon dioxide gate dielectric of current Metal – Oxide - Field - Effect - Transistors (MOSFET). These issues limit SiO_2 to be as a good gate dielectric of the next MOSFET generations [4-8].

However, the Nano particle size and Nano powder structure affect the properties of Nano composites in so how that the dried gel leads to the reduction treatment at 50°C and makes some difficulties in obtaining homogeneous and narrow size distribution of Ni Nano particles in composites with more SiO_2 content.

We have thus demonstrated a series of experiments to provide a good dispersion of Nano sized particles in a more concentrated system [2-3]. The Nano structured properties of Nano composite NiO-SiO_2 have been studied using the XRD technique. XRD spectra indicates that the crystal-particle size of the NiO-SiO_2 films is smaller than that of the Ni- SiO_2 films which have larger specific surface areas, less dangling bonds,

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DOI: 10.22051/jitl.2019.24178.1025

less surface defects, and duty porosity [1-4]. In the present work, we add more SiO_2 content from 80% NiO-20% SiO_2 to 70% NiO-30% SiO_2, and find smaller size NiO particles. By adding more SiO_2 to NiO, the obtained powder trends to crystalline structure. At room temperature, it has amorphous structure and can be therefore used as a good gate dielectric of the next nano transistor generations.

2 Experimental procedures and details

The sol-gel process is commonly applied to synthesis such as NiO materials owing to its several advantages such as low temperature processing and the ability to prepare materials in various shapes, compared with the conventional preparation procedures of glass and ceramics.

The prepared NiO-SiO_2 gel is blue. Tetraetnyl-Orthosilicate (TEOS) (Merk>99%) as the SiO_2 precursor, was hydrolyzed with di-ionized water in ethanol acts as a mutual solvent. TEOS, in ethanol was hydrolyzed with water containing Acetic acid at room temperature for 30 min before the solution was mixed with hexahydrated Nickel Nitrate ($Ni(NO_3)_2.6H_2O$)(AR.98%) with the NiO precursor at 0 ^oc in specific molar ratio to obtain various content of NiO. In another container the hexahydrated Nikel Nitrate was dissolved in water, after dissolution some Nitric acid was added. After 30 min stirring at room temperature, the sol was vibrated for 20 min in ultrasonic bath to deconglomerate particles before relaxed at room temperature for 30 min. The sol was stirred at 40 ^{0}C until it turned in to gel before removal of ethanol (about 48 hours). After gelation, samples were dried at 70 ^{0}C to remove water and Acetic acid. After that the rump samples were milled with mortar and calcinated at 50 ^{0}C. The thermal gradient during experiment procedure and the samples were put in an oven for 2 hours at calcination temperature, 50 ^{0}C. All the samples were dried in air using a furnace in order to form NiO.

The average grain size can be deduced from the scherrer equation [9]

$$D = \frac{K\lambda}{\beta\, cos\theta},$$

where D is the crystallite size of NiO–SiO_2, K is a constant (0.94), λ is the wavelength of X-ray (Cu_{ka} = 1.5406 Å), β is the true half- peak width, and θ is the half diffraction angle of the centered peak in degrees.

The phase composition of NiO-SiO_2 films is studied using the XRD technique. The X-ray diffraction pattern is obtained on a Phillips PW·1 710 X-ray diffractometer, as stated above, using Cu_{ka} radiation as X-ray source at an angle (2θ) ranging from 10-80 degrees. The measurement is carried out at a step= 0.02, exposition 15.5/step. The strongest peak of NiO–SiO_2 corresponding to (200) is selected to evaluate the crystallinity of the samples. The mean crystallite size, D, is determined from the X-ray (the peak width of half maximum) of the most intense line in the X-ray diffraction pattern

The composition and structure of the NiO-SiO_2 powder is investigated by XRD and shown in Figs 1, 2, and 3.

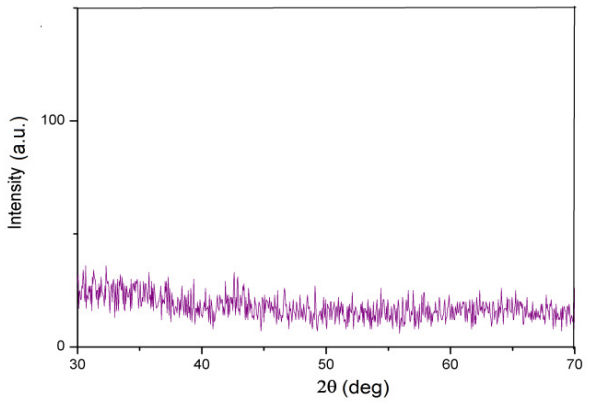

Figure 1. X-ray diffraction pattern of the 80% NiO-20% SiO_2 nanostructure at room temperature. There is no crystallite phase.

The XRD pattern of NiO – SiO2 in Fig 1, shows an amorphous phase at room temperature and crystallizes directly from amorphous to NiO and Ni FCC phases at temperatures as low as 50°C.

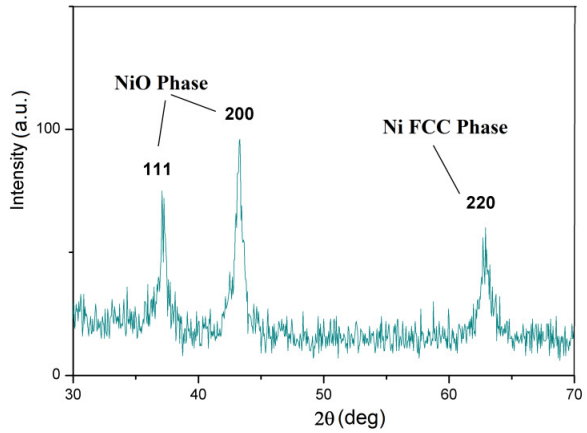

Figure 2. X-ray diffraction pattern of the 80% NiO- 20% SiO_2 nanostructure at 50°C. There are two NiO and one Ni FCC phases.

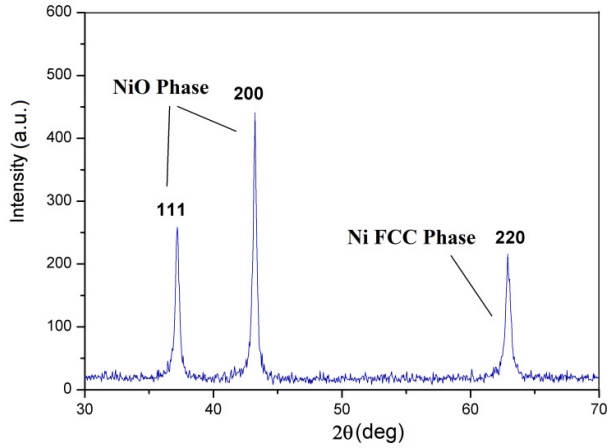

Figure 3. X-ray diffraction pattern of the 70% NiO - 30% SiO_2 nanostructure at 50°C. There are still two NiO and one Ni FCC phases.

As one can see in Figs 2 and 3, some sharper peaks are revealed which indicate (110) and (101) orientations (NiO and Ni FCC phases). By increasing the content of SiO_2 in powder mixture, there are no changes in NiO and FCC phases of Ni attributed to the lower surface energy, which is important in the nucleation of crystals.

3 Results and Discussion

Figures 2 and 3 show that all the samples are characterized by the presence of only one displayed Ni FCC phase, the superfine phase of silicon dioxide is amorphous.

The content Ni and SiO_2 in Figs 2 and 3 are 80% Ni-20% SiO_2 and 70% Ni-30% SiO_2, respectively. It is clear that an increase about 10% in the content of silicon dioxide in the samples, the particles size reduces at 500 oC.

Although the intensity of NiO and Ni - FCC phases increase significantly, the NiO/Ni intensity ratio is the same. The broadening of NiO and Ni peaks indicate that the mean crystallite size reduces for 80% Ni-20% SiO_2 to 70% Ni-30% $Si0_2$, from 30 to 18 nm (measured with Scherrer equation).

The reduction of particle size could be due to reduction of the specific surface area of the samples as found in [2] and suppressing the processes of sintering of the nano dispersed Nickel phase. The huge peak (200) and the broad peak (111) around 2θ= 43 and 36, indicates the NiO phases and (220) peak represents Ni FCC phase. To find an accurate size of particles, an X-powder method has been used and the result has been revealed in Fig 4.

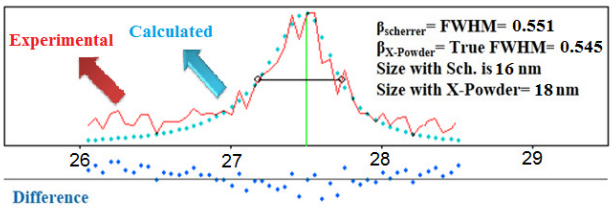

Figure 4. X - powder method has been used for determining NiO particle size.

We find that the NiO peaks get narrower and grow with respect to amorphous silica background. The nanoparticles size is obtained using the X-powder method and shows grains with 16-18 nm.

The complex dielectric function (ε) can be described [9] for spherical particles embedded in a matrix with dielectric functions ε_m and ε_p, respectively, as of the form

$$\frac{\varepsilon - \varepsilon_m}{\varepsilon + 2\varepsilon_m} = f_p \frac{\varepsilon_p - \varepsilon_m}{\varepsilon_p + 2\varepsilon_m},$$

where f_p is the volume fraction occupied by the particles, ε_m and ε_p are the dielectric functions of the SiO_2 matrix (NiO particles), which will be discussed in more details in the future. It can reduce leakage

current due to its amorphous structure (shown in Fig 1) and its high-K dielectric constancy.

4 Conclusions

There are NiO and Ni-FCC phases in Figs 3 and 4, which confirm the presence of NiO nano particles in 70% NiO-30% SiO_2 with a size of about 18 nm. Composite films can be introduced as a good gate dielectric for the future Field Effect Nano-transistor devices. The reduction of NiO particle size indicates that these nano composites 70% NiO - 30% SiO_2, could be used for gas detection as well.

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