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EFFECT OF ANNEALING ON THE MORPHOLOGICAL AND ELECTRICAL PROPERTIES OF NI NANOWIRES

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Abstract-In the present work, we report here the synthesis of Nickel nanowires within the pores of anodic alumina template via electrodeposition process. The in-situ Ni nanowires are annealed in air at 2500C for four hours. These nanowires are characterized by using scanning electron microscopy (SEM), X-ray diffractometer and source meter. SEM photographs reveal the large scale, uniform and dense growth of Ni nanowires which are in agreement with the pore diameter of 100 nm. X-ray diffraction pattern of Ni nanowires being annealed at 2500C shows the existence of NiO (111) & (200) peak in addition to Ni (111), Ni (200) and Ni (220) peaks. The current-voltage characteristics of Ni nanowires show the linear behavior whereas the annealed Ni nanowires show the non-linear behavior. The non-linear behavior may be due to substantial conversion of Ni to NiO nanowires. I-V characteristic of annealed Ni nanowires shows the increase in sensitivity towards different relative humidity levels (47%, 52%, 57%, 71%) which suggests its possible applications in humidity sensor device.
Keywords: electrodeposition, humidity sensor, nanowires, template synthesis

1. INTRODUCTION

Recently, humidity sensors have attracted great attention in the world of nano science due to their broad applications in automotive, medical, construction, semiconductor, meteorological and food processing industries and various other fields including air quality control, health science and environmental protection [1–2]. Nanostructures such as nanowires, nanotubes or nanofilms have received considerable interest in the fabrication of humidity sensors due to their high surface-to-volume ratio of atoms, excellent surface reactivity, and their ability to tailor their surface and charge transport properties. In the field of research, many humidity sensors based on FETs [3], quartz crystal microbalance [4], capacitance [5] and surface acoustic waves [6] are fabricated. Humidity detection relies on the surface reaction mechanism of water vapor molecule absorption and desorption. The major types of humidity sensing materials include organic compounds, polymer composites, ceramics, semiconductors and organic/inorganic composites. Humidity sensors based on organic polymer have many challenges due to their weak mechanical strength and poor physical and chemical stability [7]. High-quality CdS nanowires [8], silicon nanowires [9] and Nanoceria based thin films [10] are also used as humidity sensor, but the metal oxides such as ceramic metal oxides and semiconductor metal oxide are found to be a good choice as humidity sensing materials. Indeed, various metal oxide nanowires i.e ZnO nanowires [11-13],CuO/Cu_2O Nanowires[14], $CoFe_2O_4$nanowires [15], $Na_2Ti_3O_7$nanowires[16], WO_3nanowires[17], SnO_2nanowires[18] and Al_2O_3 nanowires [19] have attracted considerable attention due to their ease of fabrication, clear operating mechanism, high sensing response with good operating stability, low cost, and portability.

In this paper, we report the synthesis of Ni nanowires within the pores of alumina template through electrodeposition process & their annealing effect by annealing the Ni-filled alumina membrane in the presence of air. The morphological, structural and electrical properties are also discussed for their applications as humidity sensor device.

2. EXPERIMENTAL DETAIL

All chemicals purchased from Merck are of analytical grade with high purity were used. Anodic alumina oxide (AAO) templates of diameter 100nm, thickness 60 micron and pore density 10^9 pore/cm^2 are purchased from Whatman, USA. A three- electrode electrodeposition cell was used for the synthesis of nickel nanowires where Ag/AgCl was used as reference electrode and a Gamry Potentiostat (Reference 600) was used as dc source for electrode position at room temperature. Platinum electrode was used as counter electrode (anode). The inter-electrode distance kept was 0.5 cm. An electrolyte composed of 165 g/L $NiSO_4$ + 22.5 g/L $NiCl_2$ + 37 g/L H_3BO_3, adjusted to pH 4 with dilute H_2SO_4, was used for the electrodeposition of Ni nanowires. The deposition was carried out for 1 hr which was calculated using faraday's law of electrolysis. In order to make anodic alumina template conducting, a thin film of Au was sputtered onto one side of template using sputterer coater. This Au layer alongwith conductive Cu adhesive tape act as working electrode and provide a stable cathode for the growth of Nickel nanowires. The quality of electrodeposition depends upon several parameters such as inter-

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electrode distance, PH value, agitation, current density, temperature etc. After the deposition was over, the membrane was washed with distilled water for several times and the sample was dried in room temperature.

3. RESULTS AND DISCUSSION

3.1 SEM Characterization

The nanowires were freed from template by dissolving it with 1M NaOH solution followed by rinsing with distilled water. The morphological characterization of freed nanowires was done using scanning electron microscope (JEOL JSM 6100) at an accelerating voltage of 10KV. The SEM photograph as shown in Fig. 1 reveals the large scale synthesis of nanowires with an average diameter of 100 nm in agreement with the pore diameter as claimed by Whatman USA. SEM photograph also reveals the good quality deposition with large scale and uniform formation due to typical uniformity and arrangement of pores, which is necessary for the subsequent filling of the pores by electrodeposition.

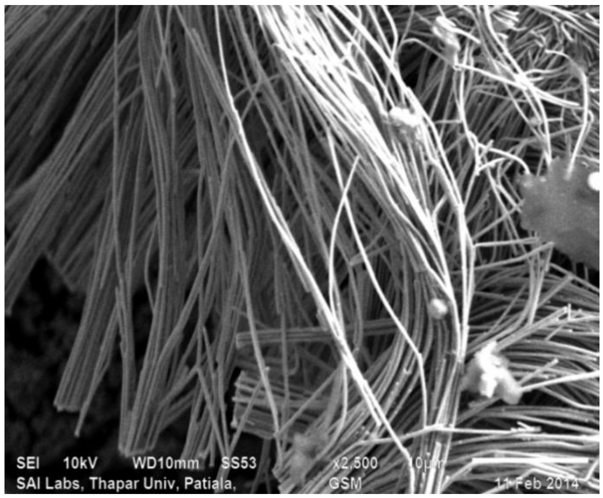

Fig. 1 Scanning electron micrograph of Ni nanowires

3.2 XRD Characterization

The crystalline phases of the as-synthesized and annealed samples were confirmed by using X-ray diffractometer (PW 1710) with Cu-Ka radiation (λ = 1.54A^0) radiation as shown in figure 2. From XRD patterns, the peaks corresponding to planes (111), (200) and (220) of Ni have been observed in case of as-deposited samples. In case of samples annealed at 250^{0}C in air for 4 hrs,, two additional peaks (111) and (200) of NiO, have also been observed. The average crystalline size (D) is determined using Debye-Scherrer formula and comes out to be 21 nm for Ni. The Debye-Scherrer formula is given by:

D= K λ/ (β Cosθ)

Where λ= 1.54 Å, K= 0.9, known as shape factor, and β = Full Width Half Maxima (FWHM).

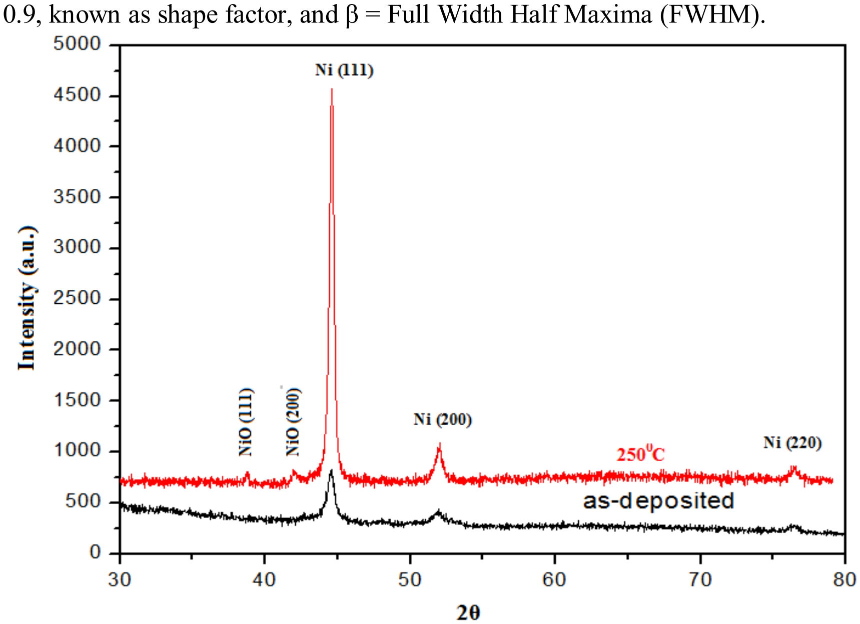

Fig 2. XRD of as-deposited and annealed (250^{0}C) Ni nanowires

XRD pattern shows the substantial conversion from Ni to NiO nanowires which may be expressed in three steps.
Step 1: Oxygen molecules absorb onto the surface of the as-prepared Ni nanowires and decompose into oxygen atoms, forming an adsorption layer of oxygen atoms.
Step 2: The atoms of the Ni nanowire lost two electrons to form Ni^{2+} ions and the oxygen atoms obtained the two electrons removed from the Ni atom, forming a negatively charged oxygen ion. This will create an electric field in the surface of nanowires; facilitating the transport of Ni^{2+} ions. The tendency to form a NiO phase is small at the beginning of the oxidation, since the localized reaction of oxygen with an underlying metal substrate exerts a very strong influence on the free energy barrier of oxide nucleation. This results in the formation of an amorphous NiO layer on the surface of the Ni nanowires.
Step 3: NiO grains formed on the surface layer of the nanowires and oxidation layer grew from the outer surface of Ni nanowires to the interior of the nanowires at high temperature with the increase of the oxidation time.

3.3 Electrical Characterization

Current-voltage (I-V) characteristics of Ni nanowires (NWs) before and after annealing were carried out using specially designed assembly (ITO/Ni-NWs/ITO) as shown in figure 3 using “Keithley Digital Source Meter” 2400. Collective I-V behaviour of Ni-nanowires follows Ohm’s Law whereas the non-linear behaviour os observed for Ni nanowires after annealing which may be substaintially conversion of these nanowires from Ni to NiO (p-type semiconductor) as shown in Fig 4 (a) and Fig 4 (b). As the material conductivity depends upon the water molecules adsorbed on it, nanowires find themselves as a suitable candidate for humidity sensor devices due to large surface area to volume ratio (active surface sites). The nanowires humidity sensors usually work on the principle of amount of water adsorbed on the nanowire surface. Adsorption generates the variation in electrical resistance with humidity which is well explained through “Grotthuss Mechanism”. As relative humidity increases conductance increases. Furthermore, the effect of humidity is also observed for annealed Ni nanowires which shows the variation in conductivity when these nanowires are exposed to different humidity levels (47%, 52%, 57%, 71%); which suggests their possible applications as humidity sensors.

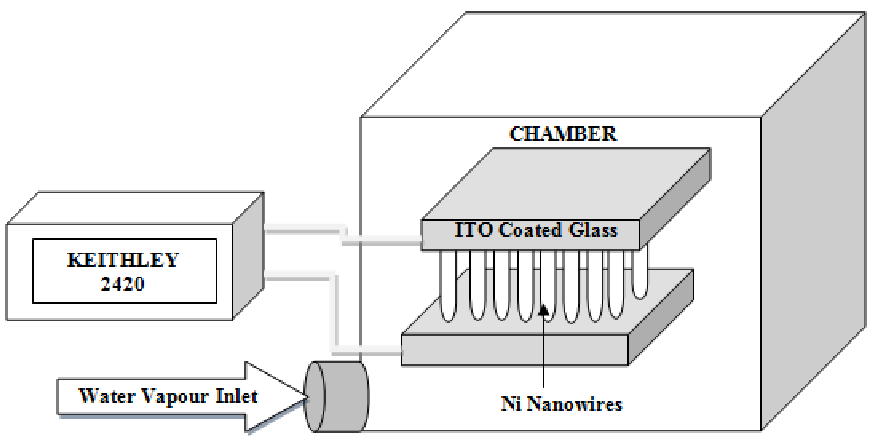

Fig. 3 Setup for I-V Characterization

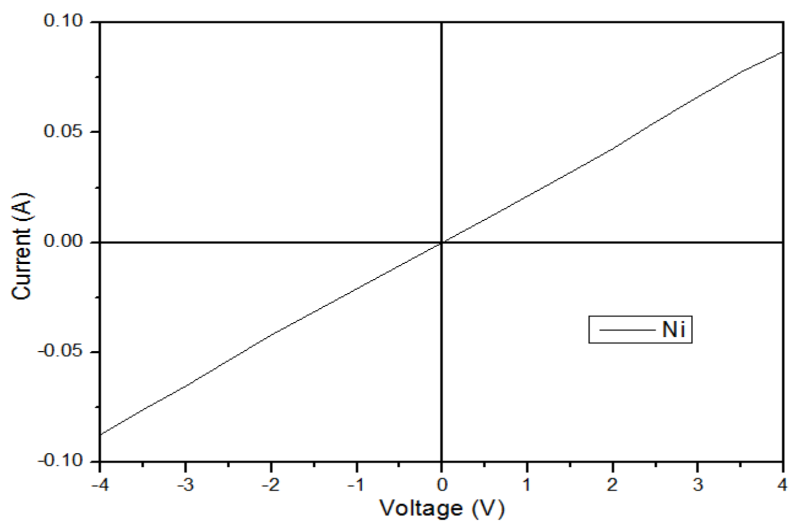

Fig.4(a): Collective I-V behaviour of Ni nanowires (as-deposited)

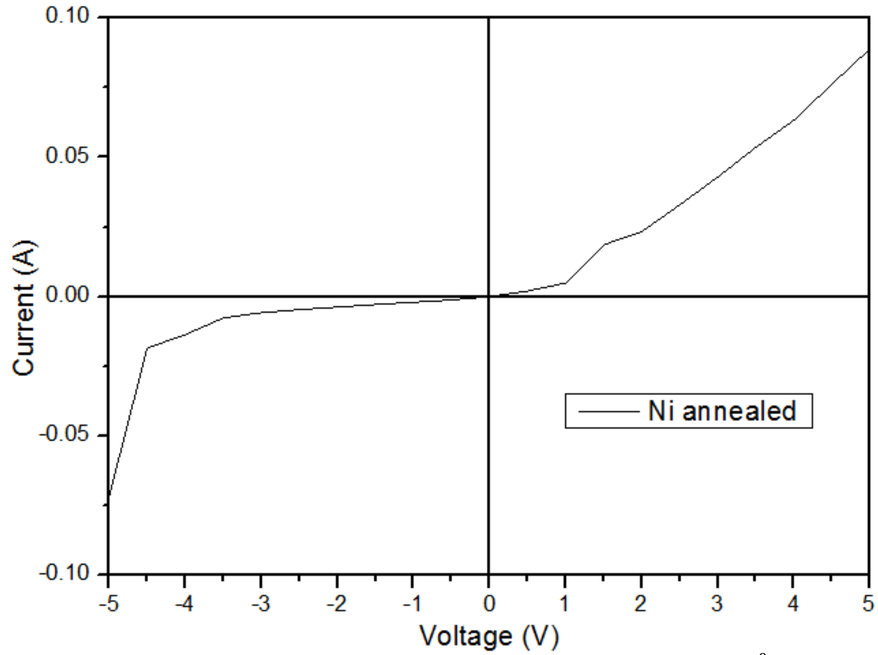

Fig.4 (b) : Collective I-V behaviour of Ni nanowires annealed in air at 250^{0}C for 4hrs

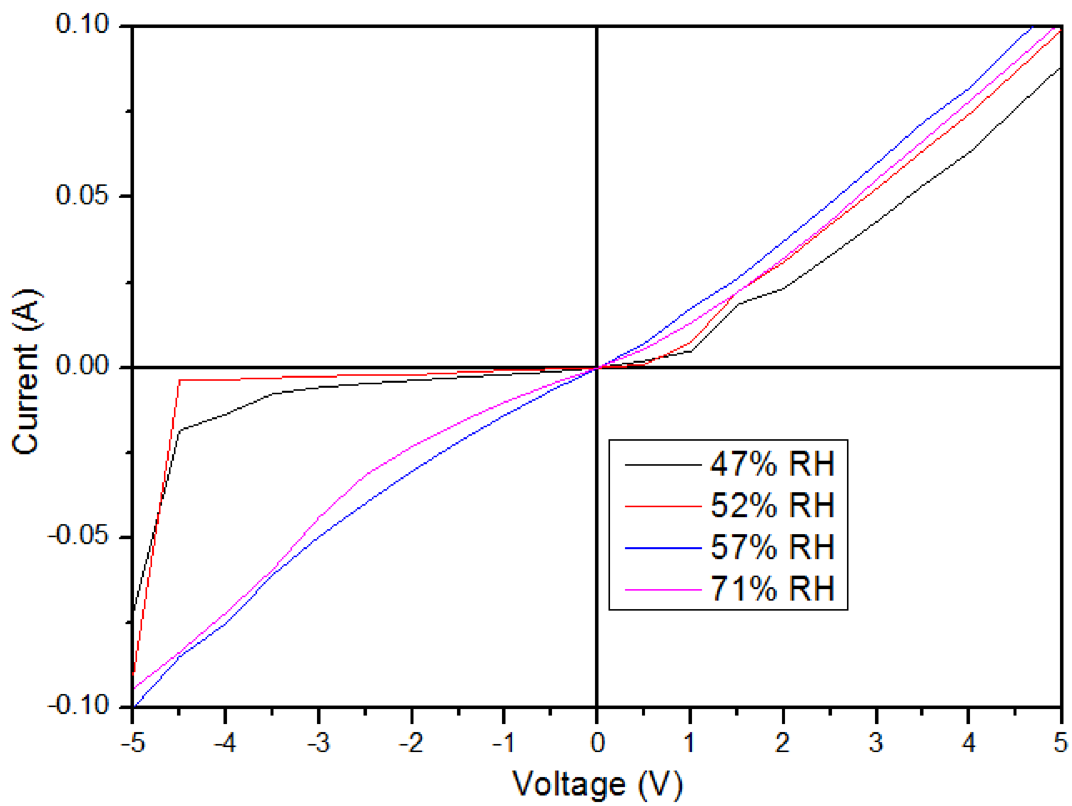

Fig.5 I-V characteristics of annealed-Ni nanowires at different relative humidity (RH) levels which suggests its possible applications as humidity sensors.

4. CONCLUSION

Template electrodeposition is an easy route to synthesize large scale, uniform and high density Ni nanowires. The in-situ annealing of Ni nanowires leads the substantial conversion of these nanowires into NiO nanowires which find their potential applications as humidity sensors. The further annealing effect at higher temperature in the presence of air and O_2 environment may be studied for their morphological studies and applications in various electronic devices.

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