Structural Study Of A-Fe_2O_3 Attached MWCNT Thin Film Prepared By Simple Chemical Method

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Abstract: *Multiwall Carbon Nanotubes (MWCNTs) were functionalized with HNO_3-H_2SO_4 acid mixture to obtain better surface properties while used for attachment of different elements or compounds. Simple chemical method was used in this work to obtain magnetite (Fe_3O_4) nanoparticles attached MWCNTs thin film. On the surface of CNTs, initially hematite (α-Fe_2O_3) phase of iron oxide was confirmed by XRD analysis. This phase of nanoparticles on the surface of CNTs was further converted to the magnetite nanoparticles by annealing at temperature 500^{o}C in H_2 gas environment. The morphology of these films with different concentration variation was analyzed with scanning electron microscopy (SEM)*

Introduction

Since Iijima's observations in 1991[1], the utilization of carbon nanotubes (CNTs) in consumer products and industrial applications has increased significantly because of their extraordinary mechanical, electrical, and optical properties[2-4]. For example, transparent conductive CNT films can potentially replace the brittle and expensive indium tin oxide films that are currently used in touch screens, liquid crystal displays, and solar cells[5-6]. The incorporation of CNTs in bicycle frames, car bodies, and windmill blades not only reduces the weight but also significantly enhances the mechanical strength of these structures[7]. Lithium ion batteries (LIBs) are becoming the main power source in portable electronic devices due to their high specific energy, superior energy density, stable cycling and less memory effect[8-9]. Recently the anode material used for commercial LIB is graphite which has low electrical potential with respect to lithium, good electrical conductivity and superior safety [10]. Still it is no longer the best candidate for anode material due to its low specific capacity and poor rate performance. During past few years many researchers has got more attention on transition metal oxides (MO where M is Fe, Co, Ni, Cu etc.) because of high theoretical specific capacity due to the conversion mechanism, which is different from the intercalation mechanism of graphite. It is observed that among these metal oxides Fe_2O_3 is one of the best materials due to high specific capacity, huge abundance, low cost and environment friendly [11-14]. But these have some drawbacks one is crumbling, pulverization and consequent disconnection with the current collector during discharge/charge process which leads to fast capacity fading and another one is the severe agglomeration of active materials, which cause the reduction of active surface area [15]. These issues may overcome mainly by two ways: one is to synthesize nanostructured Fe_2O_3 with various nanoscale morphologies and another way is to fabricate hybrid nanostructure, where the active material, Fe_2O_3, is embedded into conductive matrix (16-17). Carbon materials especially carbon nanotubes are widely applied as conductive matrix due to its unique electronic properties. So it is very important to investigate the structural properties of Fe_2O_3 nanoparticles decorated MWCNTs. In this paper we report a simple chemical method to prepare α-Fe_2O_3 attached MWCNT thin film.

Experimental

Hematite attached MWCNTs thin film were prepared by simple chemical method by using anhydrous $FeCl_3$ and acid (HNO_3-H_2SO_4) treated MWCNT. At first prepared the m molar solution of $FeCl_3$ in which added the acid treated CNT and sonicate about 2 h. Then stirring it for several hour with basic condition. Ethylene glycol is used as capping agent during synthesis. Dark black solution has put to settled down for sometimes and thin film was prepared with this solution by simple drop casting method on silicon substrate. The film was characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM).

Results and Discusssions

The α-Fe_2O_3 film formed on the silicon substrate was characterized with XRD with Cu K_α (1.5406 Å) line as the source. Figure 1 shows the XRD patterns of the α-Fe_2O_3 film.

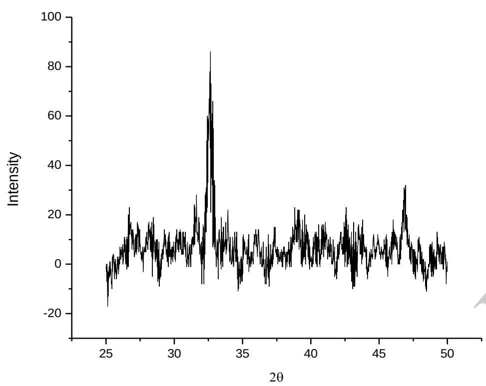

Figure. 1. XRD patterns of α-Fe_2O_3 attached MWCNT thin film formed on silicon substrate

The XRD patterns of α-Fe_2O_3 incorporated MWCNT film exhibits the prominent peak at 2θ equal to 32.6 corresponds to the diffraction plane (104). The average grain size inside the film were determined using Debye Scherer's formula, $D = 0.9\lambda/\beta \cos\theta$ where D is the crystalline size, λ is the wavelength of CuK_α line (1.5406 Å), θ is the diffraction angle and β is the full width half maximum (FWHM) of diffraction peaks in radian. The estimated particle size by XRD data is 35 nm.

Figure 2 shows the XRD patterns of the magnetite incorporated MWCNT film, obtained by heating the α-Fe_2O_3 film at 500^oC temperature in H_2 gas environment. The prominent peak of this film observed at 2θ equal to 35.6 corresponds to the diffraction plane (110). The estimated particle size is about 22 nm.

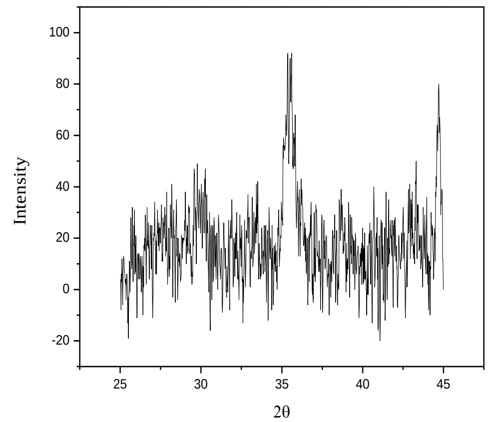

Figure. 2. XRD patterns of Fe_3O_4 attached MWCNT thin film formed on silicon substrate

Figure 3. shows the SEM image of the formed α-Fe_2O_3 attached CNT film at different iron concentration. It is clearly observed by SEM image that the hematite particles are attached on the surface of MWCNT. From the SEM image the particles are of almost uniform size of around 25 nm which is almost close to that of the XRD result.

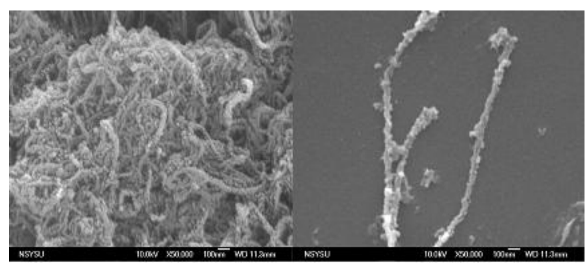

Figure. 3. SEM images of α-Fe_2O_3 attached MWCNT thin film.

Figure 4. Shows the result of the EDX for α-Fe_2O_3 attached MWCNT thin film. These spectra show the presence of major peaks from the C, O and Fe elements.

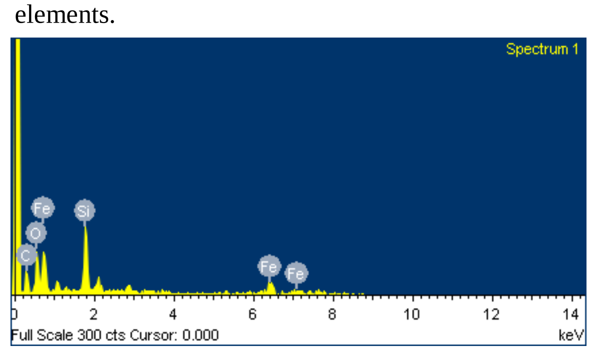

Figure. 4. EDX elemental microanalysis of α-Fe_2O_3 incorporated CNT film.

We also observe the intense peaks of Si due to the film are prepared on silicon substrate. It is observed that the film contains C, O, Fe element as 24.44, 15.43 and 50.54 Weight% respectively.

Conclusion

The α-Fe_2O_3 attached on MWCNT surface is made by a simple method. The grain size of the iron oxide inside the film is about 25 nm. Element present in the sample is C, O, and Fe confirmed by EDX.

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