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Synthesis and characterization of MWCNTs/PVDF nanocomposite and its electrical studies

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ABSTRACT

We report the electrical studies of nanocomposites of Multiwalled Carbon Nanotubes (MWCNTs) and Poly(vinylidene fluoride) (PVDF).The MWCNTs were synthesized by chemical vapour deposition (CVD) method. Solvent evaporation method is used to prepare MWCNT-PVDF composite films. Scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FTIR) were used to study structure and morphology of the composites. The temperature dependant conductivity measurements for the nanocomposites were performedusing the well established four probe method.

Keywords: Carbon nanotubes; PVDF; XRD; SEM

INTRODUCTION

Carbon nanotubes have been intensively studiedin both fundamental and applied research fields forover one decade [1]. Among the CNTs mostly used, the multi-walled carbon nanotube (MWNT) deserves special attention because of its low cost and mass production. It

has been considered to make composite of MWCNTs asfiller with conventional polymer matrix such as Poly(vinylidene fluoride) (PVDF) for flexibilityand high quality of electrical properties [2].However,the preparation of satisfactory composites is still a greatchallenge. An analysis of the registered patents regarding thefabrication of polymer composites containing carbonnanotubes reveals that several fundamental processingchallenges such as purification, dispersion, alignment andadhesion of nanotubes and the limitations in interfacial loadtransfer must be overcome[3]. In addition, another majorobstacle to use CNTs as fillers is the high expenses.The electrical conductivity is a sensitive probe of composites. Indeed, the percolation behavior in the electricalconductivity of nanotube–polymer composites has beenwidely studied for several years. The high aspect ratio ofCNTs is known to be advantageous in making a percolationnetwork at relatively small loading percentages of order 1wt% of nanotubes.[4]

In this work, we investigate the electrical performance such as temperature dependant electrical conductivity of MWNT-poly(vinylidene fluoride) composite. Structural analysis and physical performances of the samples were examined by scanning electron microscope (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and Four probe method

MATERIALS AND METHODS

In this work MWNTs were prepared by pyrolysis of acetylene over Mm based [Mm-Mischmetal, AB3 alloy hydride catalyst] using thermal CVD technique. The alloy hydrides were obtained through hydrogen decrepitation route. The as-synthesized MWCNTs contain some amorphous carbon and catalytic impurities [5].MWCNTs were first subjected to air oxidation at 450°C for 2h to remove the amorphous carbon and then refluxed with conc.HNO3 for 24h to remove the catalytic impurities.This was followed by washing with de-ionized water several times and then the sample was dried in air for 30min at 100°C. For the formation of nanocomposite film, MWCNTs were dispersed in N,N dimethyl formamide (DMF) with PVDF(Merck) by constant stirring and high power sonication.Once it dissolved thoroughly in DMF(Merck), transfer the solution on to the Petri-dish and dry it at 60°C for 10 hrs in vacutherm.[6,7]

RESULTS AND DISCUSSION

The Crystalline nature of CNTs is confirmed by XRD studies [8]. Peaks indexed to (002), (100), (101) reflects hexagonal structure (Fig 1). The presence of 002 peak in the XRD data, suggests multiwalled nature of carbon nanotubes.The X-ray pattern of (Fig 2) showed both the characteristic peaks of CNT and PVDF. From the observation of this XRD we could conclude that synthesis of the composite with CNT in PVDF was successful.

The SEM images of purified MWCNTs and MWCNT-PVDF composite were shown in fig 3 and 4.From the images we could interpret that the MWCNTs were uniformly dispersed in PVDF matrix.

FTIR spectra were used to characterize the functional groups ofpolymers and CNTs after modification (10).FTIR spectraof figure 5 shows MWCNTs and figure 6 of PVDF/CNT.IR spectra of opened CNTs the absorbance bands at 3600–3100, 1700 and 1620, and 1500 cm-1 are related to the O–H, C=O, and C=C bonds.The CNT bonds which are formed in fig 5ispresent in the figure 6 along with the polymer stretching bonds [9].Hence we conform by FTIR that the CNT/PVDF composite is formed.

The specific electrical conductivity measurements of the compositesprepared from MWCNTs-PVDF were performedusing the well established four probe method [10].Measurements were performed as a function of temperaturefrom 77 K to 300 K. Figures(7,8) represents the experimental results ofelectrical resistance as a function oftemperature for MWCNTs-PVDF composite and also its I-V characteristics.In order to know the charge tranport of the hybrid MWCNTs-PVDF composites the temperature dependant electrical conductivty measurements were done. The temperature dependant values of resistance for the composite is tabulated in table 1.We observe that the material is showing the negative temperature coefficient (NTC).As the temperature increases, the energy of the electrons increases due to the increased thermal energy available and therefore enables them to be promoted to the conduction band. This results in a greater proportion of electrons being able to move around and carry charge , then more electrons that are mobile and can carry charge, the more current a material can conduct and therefore the resistivity has decreased, hence the material exhibit a decrease in resistivity with increasing temperature.

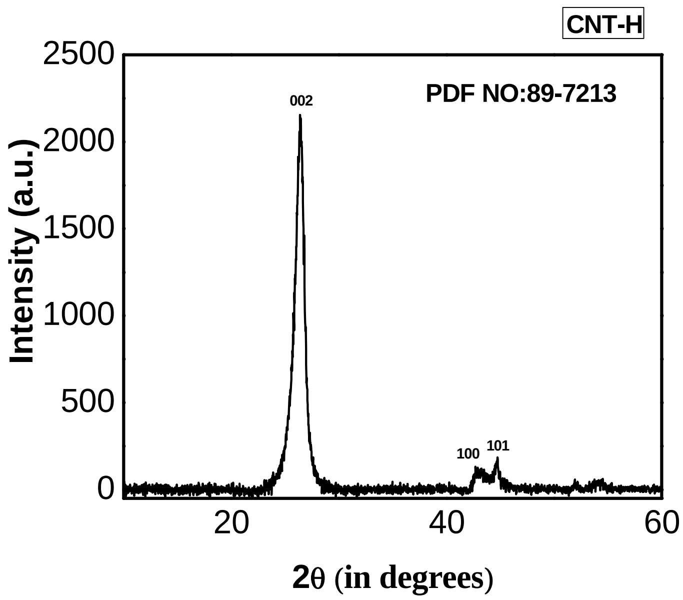

Fig.1 X-ray diffraction pattern of MWCNTs

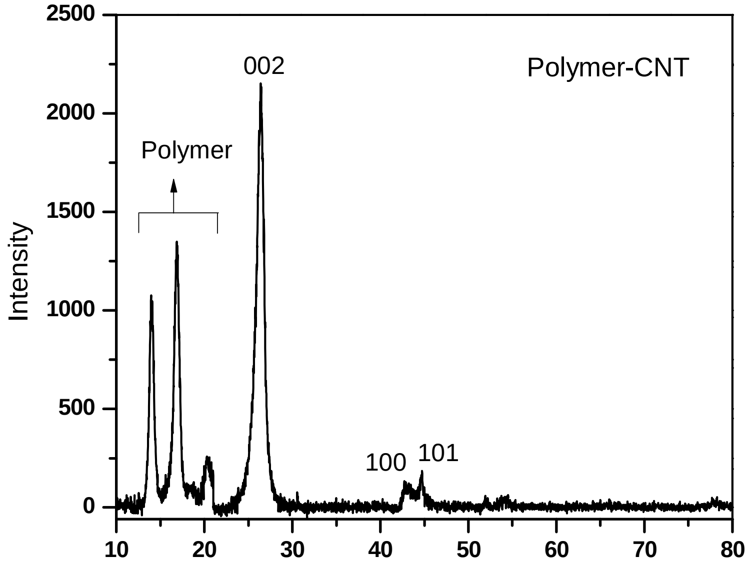

Fig.2 X-ray diffraction pattern of MWCNTs-PVDF composites

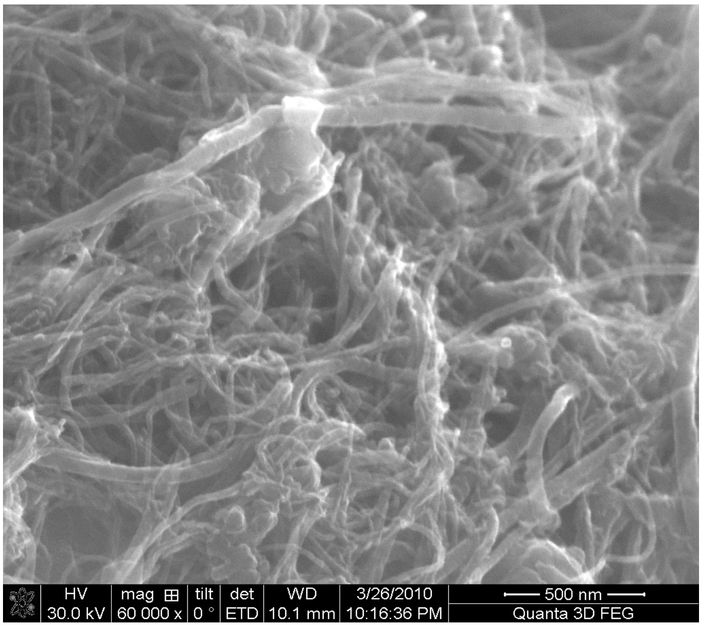

Fig.3 SEM image of MWCNTs

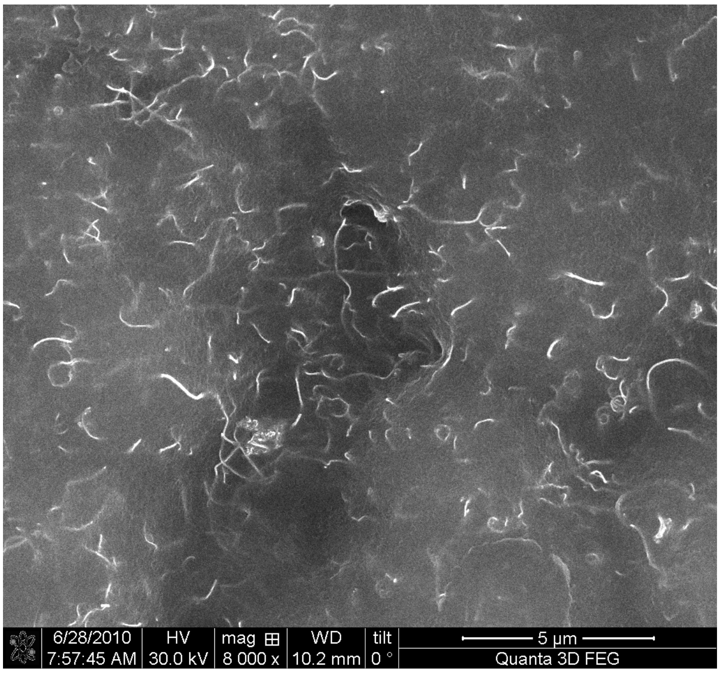

Fig.4 SEM image of MWCNTs-PVDF composites

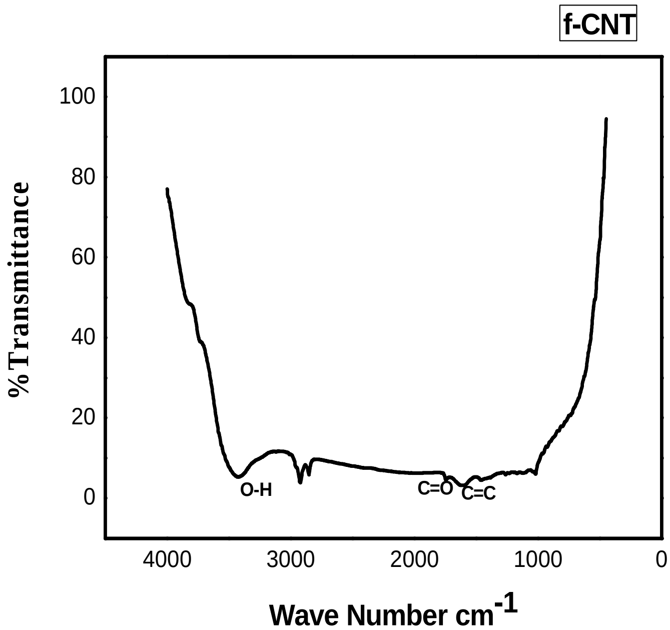

Fig.5 FTIR spectra of MWCNTs

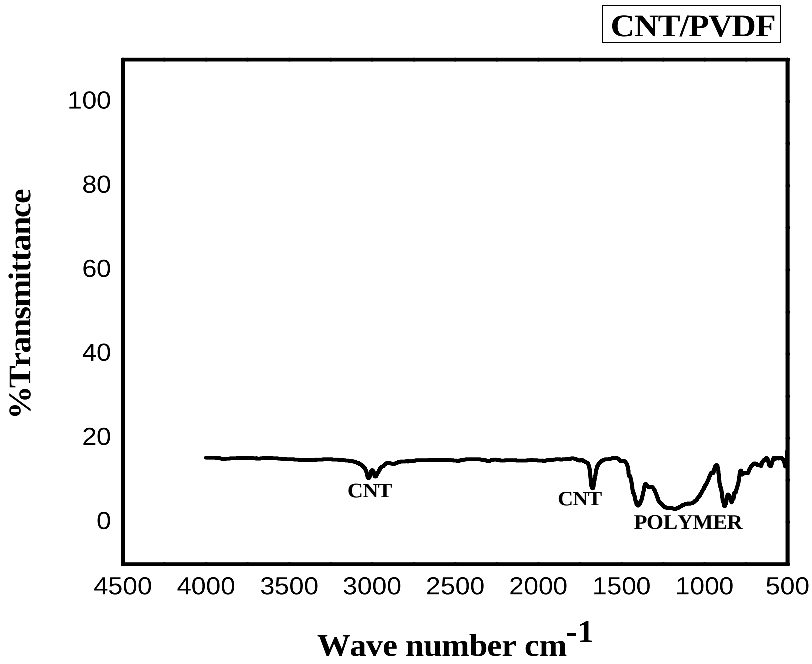

Fig.5 FTIR spectra of MWCNTs-PVDF composites

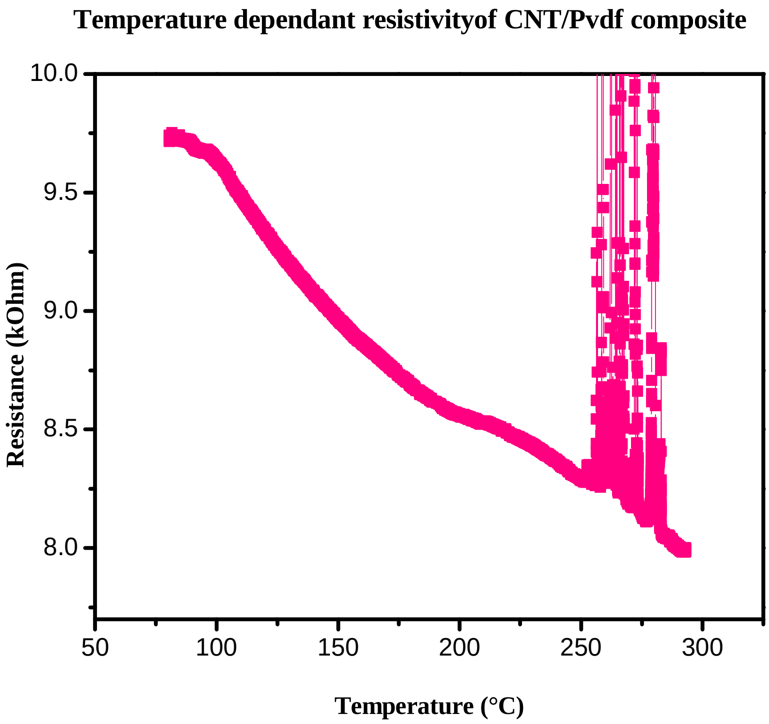

Fig.6 Temperature dependant resistivity of CNT/PVDF composite

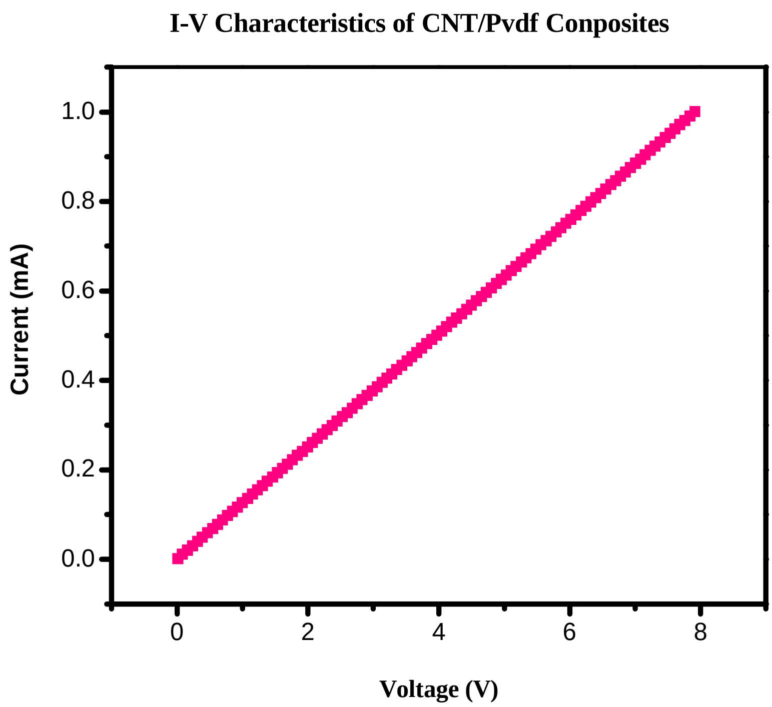

Fig.7 I-V Characteristics of CNT-PVDF composites

Table 1 [Temperature vs Resistance]

Temperature(K)	Resistance(K Ω)
79.5	9.7
157.1	8.8
224.2	8.4
278	8.13
294.7	7.9

CONCLUSION

We conclude that the MWCNTs-PVDF composite have been synthesized successfully. The crystal structure, surface morphology, presence of functional groups were studied from XRD,SEM and FTIR. The electrical properties were studied using four probe method.

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