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MORPHOLOGICAL, THERMAL, ELECTRICAL AND ELECTROMECHANICAL PROPERTIES OF POLYVINYLIDENE FLUORIDE (PVDF)-FUNCTIONALIZED CARBON NANOTUBE COMPOSITES (PREPRINT)

Varrla Eswaraiah, Krishnan Balasubramaniam, and Sundara Ramaprabhu
Indian Institute of Technology Chennai

MARCH 2012

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14. ABSTRACT
Functionalized carbon nanotubes (f-CNTs) reinforced polyvinylidene fluoride (PVDF) nanocomposites are prepared by high rate shear mixing of homogeneous suspension of f-CNTs and PVDF polymer with different weight fractions of f-CNTs. Morphology analyses show that microspheres of PVDF have been formed throughout the composite and carbon nanotubes are embedded within and in between the microspheres. As the concentration of carbon nanotubes increases in the polymer, end to end contact of the nanotubes takes place within the polymer sphere which coalesced to each other to form highly conducting path for electrons in the polymer. TGA confirms the stability of the nanocomposites as these are more thermally stable compared with base polymer. Due to reinforcement of 3 wt % of carbon nanotubes, nearly 25°C of onset decomposition temperature is achieved. DSC manifests the existence of polymer crystalline nature in the nanocomposites. Electrical conductivity results suggest that low percolation behavior has been observed in these micro-nano based polymer nanocomposites.

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Functionalized carbon nanotubes (f-CNT), multi-walled, conductivity, Strain, resistance, electromechanical properties

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Functionalized carbon nanotubes (f-CNTs) reinforced polyvinylidene fluoride (PVDF) nanocomposites are prepared by high rate shear mixing of homogeneous suspension of f-CNTs and PVDF polymer with different weight fractions of f-CNTs. Morphology analyses show that microspheres of PVDF have been formed throughout the composite and carbon nanotubes are embedded within and in between the microspheres. As the concentration of carbon nanotubes increases in the polymer, end to end contact of the nanotubes takes place within the polymer sphere which coalesced to each other to form highly conducting path for electrons in the polymer. TGA confirms the stability of the nanocomposites as these are more thermally stable compared with base polymer. Due to reinforcement of 3 wt % of carbon nanotubes, nearly 25°C of onset decomposition temperature is achieved. DSC manifests the existence of polymer crystalline nature in the nanocomposites. Electrical conductivity results suggest that low percolation behavior has been observed in these micro-nano based polymer nanocomposites. The electromechanical results shows the changes in the resistance of carbon

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nanotube-PVDF composites occurs with the application of strains due to changes in the electronic structure of CNTs embedded in polymer.

Introduction

The discovery of carbon nanotubes (CNTs) by Iijima[1] in 1991 and realization of their unique electrical,[2] mechanical,[3] thermal properties,[4] many scientists are fascinated to make future advanced composite materials including CNTs filled polymer composites as a conductive filler in polymers. CNTs are much effective due to its large aspect ratio than traditional black to make conductive polymer composites.[5] These conducting polymer composite films find a variety of applications in EMI shielding,[6] super capacitors,[7] resistive type touch sensors,[8] and transparent conductive coatings in solar cells,[9] high strength composites,[10] biosensors,[11] and fuel cells.[12] And also carbon nanotubes are being investigated as a means for large scale inspection of composite structures. Several studies suggest that CNTs possesses excellent electrical conductivity and piezoresistivity and it has been proposed that CNTs could be delamination resistance but also enable to structures with multifunctional capability. Structural health monitoring (SHM) of air frame structures is very much essential in mechanical, aerospace, civil, marine and agricultural systems. Traditional strain sensors are based on Constantan, nickel-chromium alloy offer wide static, dynamic and temperature ranges but they can measure strain only at specific locations and in fixed directions. Li et al.[13] reported that SWNTs film can be used as strain sensor at the macro scale due to the dependence of the electrical properties of the SWNTs film on mechanical deformation on nanosclae. and also Prasad Dharap et al. reported that there is a change in voltage linearly when it is subjected to tensile strain and they proposed multiple location strains can be measured by the isotropic carbon nanotube film.[14] They suggested potential of CNT films in measuring strain at the macro level. Even though they suggested SWCNTs can

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be used as strain sensor but it suffers from its high cost and difficulty in synthesis of large scale production of SWCNTs. Paulson et al.[15] studied the response of the multiwalled carbon nanotubes to mechanical strain applied with atomic force microscope. Polyvinyliedene fluoride, one of the best fluoropolymer in thermoplastic polymers which offers wide industrial applications due to its highest chemical resistance,[6] high temperature tolerance and due to its availability of polymorphs.

Experimental Part

Materials

Multiwalled carbon nanotubes are prepared in our laboratory [16] which is having a diamter of 30-35 nm and legnth of 5 μm. The polymer matrix used in this work was poly(vinylidene fluoride) powder obtained from Alfa Aesar, Ward Hill, MA. Dimethyl fomamide (DMF) was obtained from RFCL limited, New Delhi. Sulphuric acid (98 % H_2SO_4) and nitric acid (69 % HNO_3) in analytical grade were purchased from Ranbaxy, India. Millipore water was used for washing purposes. Electrically conducting silver paste was purchased from CMR direct, Cambridge, UK. EPX Plus II Applicator was used for adhering composite film over Al specimen. Aluminium specimen of 250x50x3 mm^3 was used for electromechanical studies. N-N dimethyl formamide was used as solvent for carbon nanotubes and polymer. Millipore water (18.2 MΩ) was used for washing purposes.

Synthesis of functionalized carbon nanotubes (f-CNTs)

Carbon nanotubes (CNTs) were synthesized by chemical vapor deposition (CVD) technique using misch metal (approximately 50% cerium and 25% lanthanum, with small amounts of neodymium and praseodymium)-based AB_3 alloy hydride catalysts. The as-grown CNTs not only contain pure CNTs but also amorphous carbon, fullerenes, and other metal catalysts. In

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order to remove these catalytic impurities and amorphous carbon, air oxidation was performed at 350°C for 4 h followed by acid treatment in concentrated HNO_3 for 24 h. Further it was washed with ethanol and millipore water several times in order to remove impurities. After purification, CNTs were functionalized with 3:1 ratio of H_2SO_4 and HNO_3 at 60°C for 6 h in order to impart hydroxyl and carboxyl functional groups over the side walls. After acid treatment for 6 h, the solution has been taken out from the round bottom flask and washed several times with deionized water and ethanol to remove undissolved chemicals. Finally the solution was filtered through 0.2 µm cellulose nitrate filter paper and several times this process was repeated using vacuum infiltration set up to adjust the pH of the outcome water. The final solid sample was collected from filter paper and dried at 70° C for 6 h.

Synthesis of f-CNTs reinforced microsphere PVDF nanocomposites

Using shear mixing cum solvent casting method, polymer nanocomposite films has been prepared. The synthesis of f-CNTs in PVDF prepared in the form of a thin film can be briefly explained as follows. Fixed quantity of f-CNTs as well as appropriate amount of PVDF was dispersed separately in dimethyl formamide (DMF) with the help of ultrosonicator for 1 h at room temperature for the preparation of fixed wt% f-CNTs in polymer matrix. These two solutions were mixed together by ultrasonicating for 1 h and the mixed solution was transferred into shear mixer and stirred at room temperature at 4000 rpm for 2 h and at 80°C for 30 min. The stable dispersions of the composite solutions were obtained after shear mixing. The composite solution was taken out from the mixer and transferred into petri dish and kept in an oven at 80°C to remove the solvent. Automatically peeled off films were collected and cut into required dimensions for further measurements.

Characterization

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The powder XRD measurements were performed with a PANalytical X'Pert Pro X-ray diffractometer with nickel-filtered Cu Kα radiation as the X-ray source. The pattern was recorded in the 2θ range of 10°-90°. Functionalization of the CNTs was confirmed by Fourier Transform Infrared Spectrometer (Spectrum one, Perkin Elmer, USA). Minute quantity of the sample was mixed with potassium bromide and pressed in the form of pellet using hydraulic machine and the spectra was recorded for 3 times and average was used. To observe the morphology of the synthesized samples, FESEM (Quanta 3D) was performed. Powder was attached to the conducting double sided carbon and pasted over Aluminium specimen and FESEM was operated at different voltages (10-30 kV) to get good contrast image. Thin films of the composites were cut into small pieces and attached to the specimen and micrographs were recorded. DC room temperature electrical conductivity of the composite films was characterized by standard four probe point technique in order to eliminate the contact resistance with Keithley 2400 source meter and keithley 2182 nanovoltmeter. Silver paste was used for electrical contacts to the sample. High resistance of the composite films was measured using keithley 617 programmable electrometer with two probe technique. Thermal decomposition of the polymer nanocomposites has been evaluated by recording weigh loss of the composites in Air atmosphere with 10°/min using NETZSCH instrument. Crystalline nature of the polymer and carbon nanotube-composites has been evaluated by the differential scanning calorimetric spectrometer in Nitrogen atmosphere with 10°C/min. Electromechanical properties of the polymer composites were studied with Materials Testing System (MTS 810, USA) combined with source meter (Keithley 2400). Resistance data collected simultaneously from the composite film while the film undergoes tensile and compression test using GPIB interface (National instruments, USA) by custom made Lab VIEW programme. On one side of the specimen, metallic gage was attached and on another side composites film was pasted using high strength epoxy. Strain gauge was attached on other side of the Al specimen. Resistance data was acquired from the composite film from

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keithley 2400 source meter using Lab VIEW programme by attaching two copper leads using conducting silver paste.

Results and Discussion

Figure 1a shows the powder X-ray diffractograms of functionalized multiwalled carbon nanotubes (f-CNTs), polyvinylidene fluoride (PVDF) and 2 wt% f-CNTs/PVDF composite. It can be seen that pure PVDF membrane is crystalline in nature with visible peaks at 18.6° and 20°. The sharp peak at 20° can be attributed to the presence of β-polymorph. Figure 1a also shows the XRD pattern of f-CNTs and it contains a sharp peak at 26°, which is indexed (002) plane of hexagonal graphite.[16] The XRD of 2 wt% f-CNT/PVDF composite also shown in figure 1a which clearly exhibits the crystalline nature of PVDF and a small peak at 26° can be due to the f-CNTs. Since the composite contains 98% polymer and 2 wt% f-CNTs, we did not observe much intense peak from carbon nanotubes. And also there is not much difference in FWHM of CNTs and f-CNTs, that's why we did not show in the manuscript.[17] Figure 1b show the thermo gravimetric weight loss curves for different weight fractions of carbon nanotubes reinforced PVDF nanocomposites. It was observed that PVDF was stable up to a temperature of about 440°C and a nearly 3 wt % loss observed may be due to moisture. Actual decomposition starts above 450°C, leading to the formation of hydrogen fluoride and monomers within a span of next 30° C. Nearly 50 % weight loss has been observed from 450°C to 480°C. As the temperature is increased further, CNTs starts decomposing. Figure 1d shows the DSC melting curves for pure PVDF and polymer nanocomposite with 1 and 2 wt. % carbon nanotubes. In almost all cases, melting peak is sharp and indicates the highly crystalline nature of PVDF and its nanocomposites except in the case of 2 wt% CNT-PVDF nanocomposites, there is a 3°C temperature difference in

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melting point compared with pure PVDF. The addition of CNTs to the PVDF did not change melting temperature of the base polymer.

For polymer nanocomposites, the introduction of f-CNTs remarkably improves its conductivity. A rapid increase in the electrical conductivity of composite materials takes place when the conductive filler forms an infinite network of connected paths through the insulating matrix. The filler volume content under this transition state is called the percolation threshold. The electrical property of the insulating polymer can be altered by adding different carbon nanostructures, and hence the property can be enhanced and controlled by properly choosing the shape and concentrations of the nanofillers.

Figure 3 shows the electromechanical properties of the 2 wt% f-CNTs/PVDF nanocomposite. Figure 3a depicts the load and displacement curve with respective time. Load was increased monotonically and the corresponding displacement also shown in figure which clearly shows that, when the specimen is under tension, initially changes in the displacement are linear within elastic region and start going to plastic region with further increment in load. For testing electromechanical properties of these f-CNT/PVDF composites, while the specimen undergoes tensile test, simultaneously voltage was measured between two probes attached to the composite film by supplying constant current. Figure 3b and 3d are respective changes in the voltage while tensile loads are applied continuously. In the elastic region of the 2 wt% f-CNTs/PVDF composite stretching 0.17 mm of displacement, ~70 μV of change in voltage was observed. Similarly for 0.31 mm displacement of the specimen, ~182 μV of voltage change has been observed in the elastic region. When the specimen undergoes tensile test beyond elastic limit about 1mm displacement of 14.65 kN applied load, there is change of 1536 μV in voltage and still increases drastically with further increment in load and finally it break up due to permemanet damage to the specimen. Fatigue test of the 2 wt% f-CNT/PVDF composite is shown in figure 3c and 3d and also it shows the electrical properties of the composite while specimen undergoes tensile-compression cycle tests with 0.1 Hz frequency

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from 5-10 kN region in the elastic limit. Periodically, 0.26-0.44 mm displacement was observed periodically and corresponding change in voltages were 0.40-0.57 mV throughout the elastic region. Figure 4 shows room temperature DC electrical conductivity of the PVDF composites as a function of mass fraction of f-CNTs. The conductivity of pure PVDF polymer is ~10^{-15} S/m. A drastic improvement in the conductivity after adding 2 wt% f-CNTs in PVDF matrix can be seen, which may be due to the formation of conductive network in the composites. The measured conductivity of the 2 wt% f-CNTs/PVDF is about 10^{-1} S/m. Nearly 14 orders of magnitude increase with respect to pure polymer conductivity indicates the achievement of low percolation threshold in PVDF composites. In order to accomplish conduction in filled polymer systems, conductive pathways of filler particles are required throughout the polymer matrix in order to allow electrons to move freely throughout the material. Percolation theory quantitatively relates the electrical conductivity of the composite to the volume fraction of the filler. The critical volume fraction v_c, also called the percolation threshold is the lowest concentration of filler that could form continuous conductive pathways throughout the polymer matrix.

At the onset of the percolating network the electrical conductivity obeys power law

$$\sigma = \sigma_o (v - v_c)^t$$

Where σ is the composite electrical conductivity, t is the critical exponent and ν is the volume fraction of the filler, v_c critical volume fraction. As shown in the figure 6, f-CNTs /PVDF composites conductivity exactly fits with the above equation and for $v_c =$ 0.5 %, the straight line has a critical exponent t =1.97 and a correlation factor of 0.935 indicating low percolation behavior in f-CNTs reinforced polymer composites. Percolating network showing the electrical conductivity obeys power law in f-CNTs /PVDF. If there is not a CNT-CNT contact, once electrons travel from one end of the tube to another end of it will find huge resistance of the polymer so that it will not cross the barrier. When sufficient concentration is there, CNTs are starts forming network and connectivity in the matrix so that drastic rise in

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conductivity was observed in the composite. It clearly shows that all CNTs are touching each other or very close (>2 nm in distance) so that electrons can travel easily from one tube to another tube without any obstacle (through tunneling). This is known as percolation behavior in the composites and at which it reaches percolation threshold. As the concentration of CNTs in the polymer matrix increases beyond percolation threshold there will not be a much increase in the conductivity because of saturation.

Conclusion

CNTs reinforced polyvinylidene fluoride nanocomposites have been fabricated by shear cum solvent casting method. TGA results suggest that ~25°C has been improved in onset decomposition temperature of 3 wt% f-CNTs/PVDF nanocomposite with respective to pure PVDF polymer. Electron microscopy analyses suggest that microspheres of polymer have been formed throughout the composites and carbon nanotubes are homogeneously dispersed within the polymer 3D scaffold with end to end connections in the polymer. Carbon nanotubes can be utilized as networks of sensors when properly dispersed in fiber composites. The conductivity. A large increase in the conductivity of pure PVDF (~10^{-15} S/m) is acheived with 2 wt% f-CNTs in PVDF matrix (10^{-1} S/m) due to the formation of conductive network in the composites. The electromechanical results shows the changes in the resistance of carbon nanotube-PVDF composites occurs with the application of strains which can be used for the analysis of damage accumulation during testing with the integration of electrical resistance measurements with strain and load.

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Keywords: carbon nanotubes, thermal, PVDF, electrical conductivity, electromechanical.

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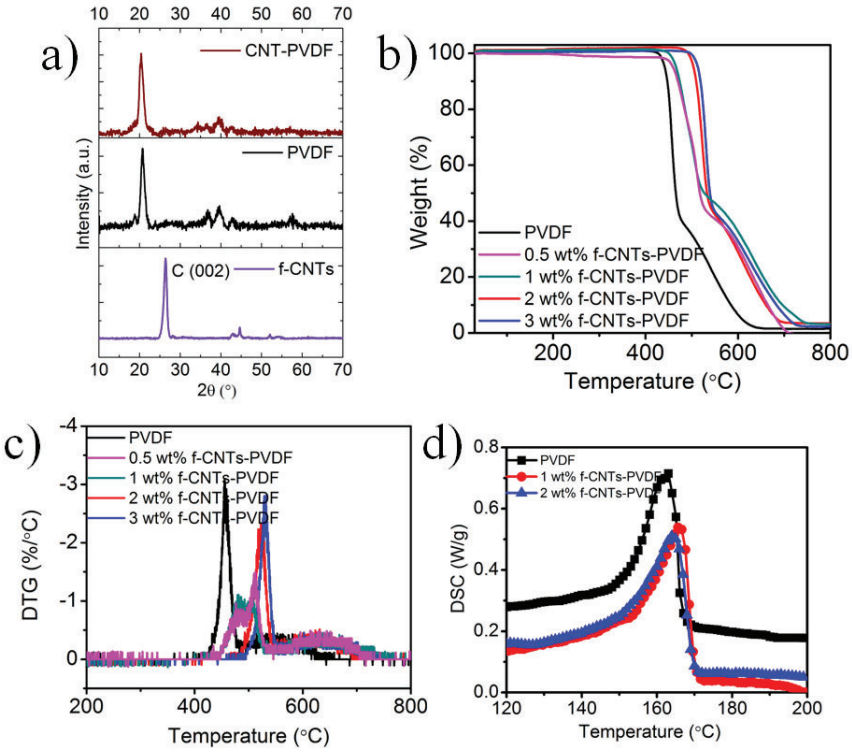

Figure 1: a) Powder X-ray diffractograms of PVDF, CNT-PVDF and CNTs, b) Thermo gravimetric (TG) curves of PVDF and its CNT composites, c) Differential TG of PVDF and its CNT composites and d) DSC of PVDF and its CNT composites.

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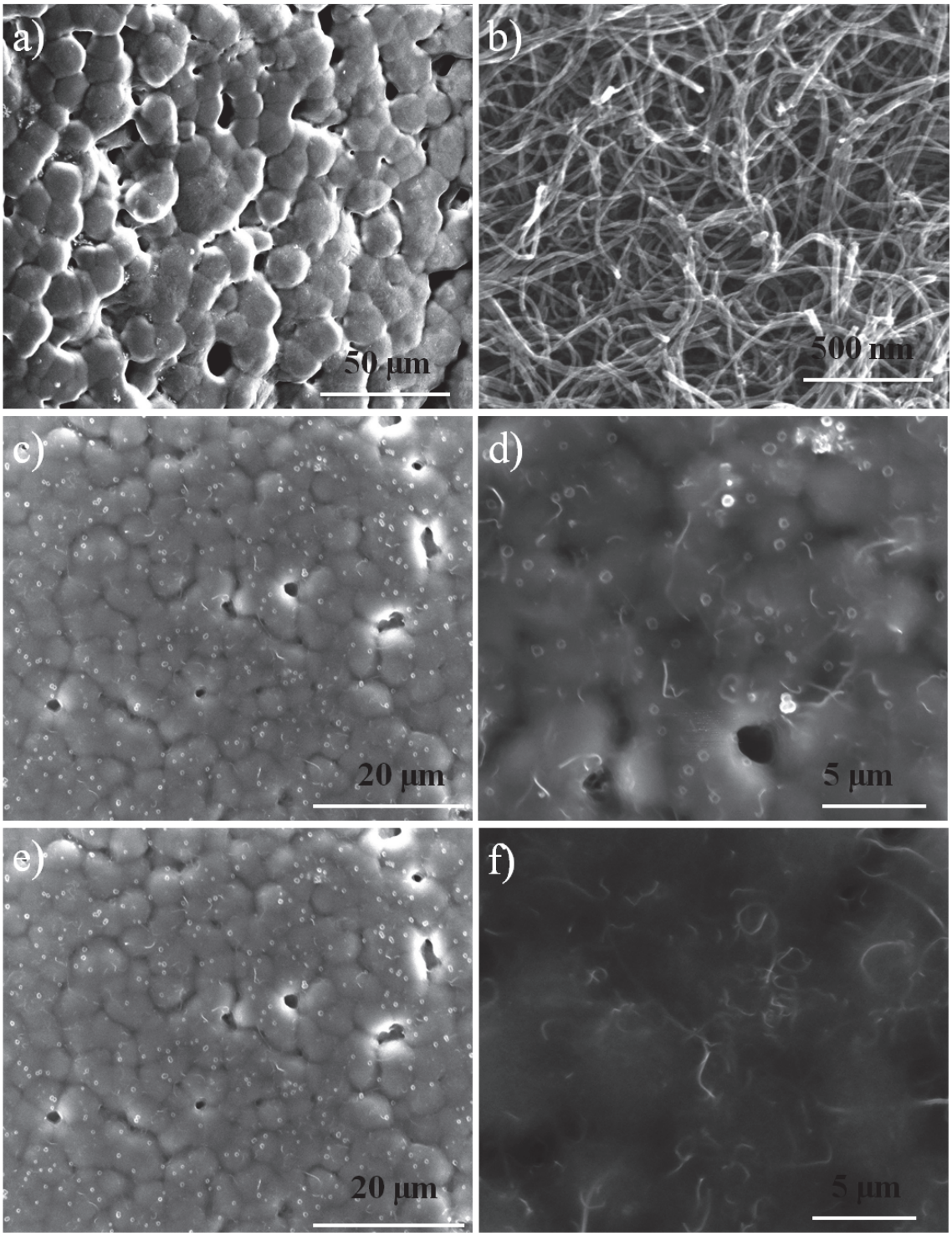

Figure 2. Field emission scanning electron micrograph (FESEM) of a) PVDF, b) Carbon nanotubes, c) 2 wt% f-CNTs-PVDF composite shows the formation of microspheres of PVDF d) high resolution FESEM of 2 wt% f-CNTs-PVDF composite confirms the embedding of carbon nanotubes in PVDF microspheres and e-f) 3 wt% f-CNTs-PVDF composite and its high resolution image.

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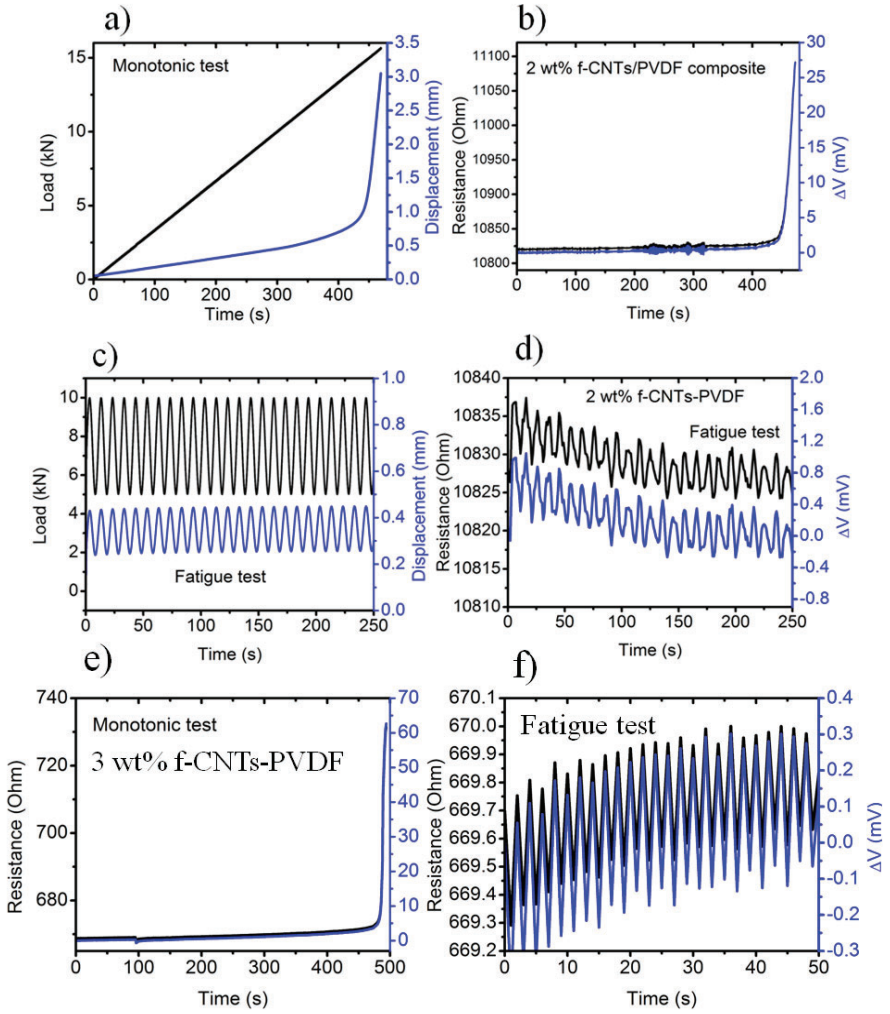

Figure 3. Monotonic and fatigue test over 2 and 3 wt% f-CNT-PVDF nanocomposites and its electromechanical response.

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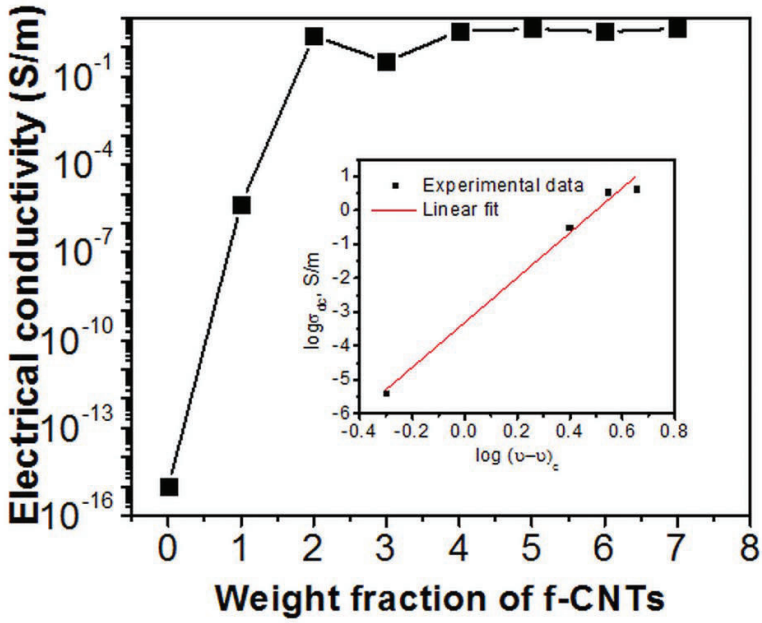

Figure 4: DC electrical conductivity of functionalized carbon nanotubes reinforced PVDF nnaocomposites.

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Nanocomposite films of microsphere polyvinylidene fluoride (PVDF) and functionalized carbon nanotubes (f-CNTs) were prepared by high rate shear mixing of homogenous solutions of PVDF and f-CNTs. Morphology of these nanocomposites exhibits the well distribution of carbon nanotubes in the PVDF. Greater onset decomposition temperature ~25°C more has been observed compared with neat PVDF. 15 orders of magnitude in electrical conductivity has been improved by addition of 2 wt% of functionalized carbon nanotubes. The electromechanical results shows the changes in the resistance of carbon nanotube-PVDF composites has been observed with applied small strains and it can be due to change in the electronic structure of CNTs.

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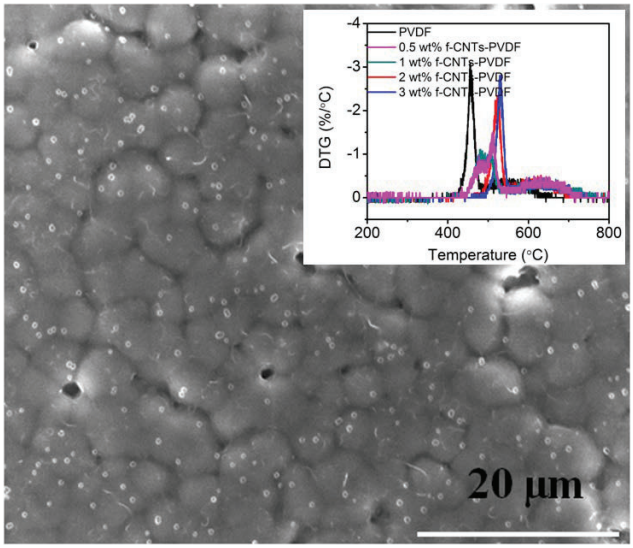

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