CHAPTER 6

RESULTS AND DISCUSSIONS

6.1 MORPHOLOGICAL CHARACTERIZATION

The SEM is one of the most versatile instruments available for the examination and analysis of the microstructure morphology of the conducting surfaces. Fig. 6.1 (a-e) shows the SEM image of different wt% of CNF doped PVDF electrospun fibers. As seen from Fig. 6.1(a-c), 0.05%, 0.16% and 0.4% CNF doped PVDF electrospun fibers indicate the absence of smooth surface.

Fig. 6.1 (d-e) clearly shows smooth and uniform surface morphology of the 0.75% and 1% CNF doped PVDF electrospun fiber unlike Fig. 6.1 (a-c). This micrograph shows linear grains, and they are uniformly distributed in the PVDF fibers. It is observed that the linear region with the size of 1–10 μm is responsible for the high conductivity of the sample, and these membranes show dense fiber structure. This increased number of linear regions leads to entrapment of large volumes of the CNF in the PVDF accounting for the increased conductivity. The interconnected linear regions in the membranes helped in absorbing liquid electrolytes and hence, the ionic conductivity of the membranes is enhanced.
Fig. 6.1  SEM Analysis of PVDF-CNFnanofiber mat  (a) with 0.05 wt.% of CNF, (b) 0.16 wt.% of CNF, (c) 0.4 wt.% of CNF, (d) 0.75 wt.% of CNF and (e) 1 wt.% of CNF
Fig. 6.2 SEM Analysis of PVDF-CNT nanofiber mat (a) with 0.05 wt.% of CNT, (b) 0.16 wt.% of CNT, (c) 0.4 wt.% of CNT, (d) 0.75 wt.% of CNT and (e) 1 wt.% of CNT
Fig. 6.2 (a-c) shows the SEM images of different wt% of MWCNT doped PVDF electrospun fibers. As seen from Fig. 6.2 (a-c), 0.05%, 0.16% and 0.4% MWCNT doped PVDF electrospun fibers indicate that there are no smooth surface and individual nano tubes. Fig. 6.2 (d-e) clearly shows smooth and uniform surface morphology of the 0.75% and 1% MWCNT doped PVDF electrospun fibers which contain several individual CNTs in the obtained micrographs unlike Fig. 6.2 (a-c). This indicates the lack of agglomeration and presence of a good dispersion. This micrograph shows the individual MWCNTs, and they are uniformly distributed in the PVDF fibers. It is observed that the linear region with the size of 1–10 μm is responsible for the high conductivity of the sample, and these findings suggest that the presence of MWCNT induces the ionic conductivity in samples.

6.2 XRD PHASE CHARACTERIZATION

PVDF is used in a wide range of applications and is particularly chosen due to its piezoelectric properties. Its crystalline morphology, in particular the presence of polar β phase crystals, is of crucial importance towards the material’s piezoelectricity. In order to identify the strength of piezoelectric property of CNF doped PVDF, samples were subjected by XRD analysis.

The β-phase is the desirable phase due to its piezoelectric characteristic. β phase has a planer TTTT (all trans) zigzag chain conformation which has space group Cm2m (orthorhombic, a= 8.58 Å, b= 4.91 Å, c= 2.56 Å) (Lando et al. 1966 and Hasegawa et al. 1972). In the present work, the different weight percentage of CNF like 0.05%, 0.16%, 0.4%, 0.75% and 1% wt were doped to identify the β phase.
Fig. 6.3 XRD Pattern of CNF-PVDF electrospun fiber mats

Fig. 6.3 shows the X-ray diffractograms pattern of PVDF/CNF nanocomposite in the range between 5–100°. As seen from Fig. 6.2, the CNF 0.05 wt.% composition in the fiber mat shows the peaks at 20.6 (200) and 20.8 (110) confirm the existence of maximum β-phase in PVDF/ CNF. Hence, XRD measurements prove that the presence of CNF is the main reason for inducement of β phase formation in nano composite. Similarly, at 0.16 wt. % of CNF and 0.4 wt. % of CNF shows that the peaks at 20.7 (200) and 20.8 (110) confirm the existence of maximum β-phase in PVDF/ CNF. Also, fig. 6.3 shows that the peaks 20.6 (200) and 20.8 (110) indicate that the presence of β phase in 0.75 wt.% of CNF which implies the significant strength of increasing piezoelectric conductivity in CNF doped PVDF.

At the same time, especially 1%wt CNF doped PVDF is the strong evidence for the existence of β phase in PVDF fibers due to improper
homogeneous mixture. Hence, XRD data shows the influence of CNF on PVDF indicating an enhancing role of CNF on β phase crystals formation in solution compounded PVDF films. Therefore, β-phase crystallization is significantly accelerated in the PVDF due to 0.24%, 0.45% and 0.75% of CNF.

![XRD Pattern of CNT-PVDF electrospun fiber mats](image)

**Fig. 6.4 XRD Pattern of CNT-PVDF electrospun fiber mats**

A nanocomposite of PVDF and CNT would be an interesting material not only because of the enhancement of piezoelectric and conductive properties of PVDF and CNT, respectively but also, due the greater β phase formation, the electrical conductivity is further increased. Therefore, it could be used as a sensing element for aircraft structural health monitoring.

Current scientific reports show the possibility to enhance and stabilize the β phase up to 100% (pure β-phase) in PVDF-CNT composites
with proper modification of CNTs. High proportions of polar β-crystalline phase ensure excellent piezo and ferroelectric activity with high spontaneous polarization. Hence, in order to identify the strength of piezoelectric property of CNT doped PVDF, samples were subjected by XRD analysis. The XRD study was analyzed for different wt% of CNT doped with PVDF such as 0.05%, 0.16%, 0.4 %, 0.75% and 1wt %.

Fig. 6.4 presents typical XRD pattern of the different conformation of pristine PVDF. Sonication of the PVDF-CNT solution composite plays an important role in formation of β-phase (Levi et al. 2004). In sonicated PVDF/CNT composite, a new crystalline peak has been observed in Fig. 6.4 of different wt.% of CNT at 2θ=20.6° that has been assigned to (110) and (200) reflections of β-phase. When the wt.% of CNT at 0.75, the peak at 20.7° shows the strong evidence of β phase. Where else at 1 wt.% the strength of the peak has reduced.

Similarly, as seen from the XRD spectrums of CNT/PVDF uniaxially stretched film, the peaks at 20.7°, 36.6°, and 56.9° are assigned to (110, 200), (020, 101), and (221) reflections of β-PVDF crystal plane, respectively (Esterly et al. 2004). The orthorhombic unit cell of crystalline β-PVDF has been indexed as a = 8.58 Å, b = 4.91 Å, c = 2.56Å (space group Cm2m).

It is found that CNTs can be wrapped by PVDF and formed into spherical particles with a diameter of several micrometers after sonicating PVDF/CNT mixture solution. From the experiment, it could be concluded that all trans chains of PVDF absorbed on the CNT surface more tightly than trans gauge chains and due to sonication of some trans-gauge –type polymers, chain can be transformed into all trans type in the composite. The all-trans molecular chains absorbed on the CNTs surface can act as nucleating agents owing to a strong confining effect of CNTs, which result in the formation of
β-phase during the crystallization of PVDF. With further increase in wt%, CNT reduces the amount of β-crystal in composites (Huang et al. 2010). This result suggests that MWCNT addition increases crystalline property of material slightly through enhanced nucleation, and thus enhanced the β-phase crystalline formation in PVDF-CNT leads to strain sensing through electrical conductivity.

6.3 MECHANICAL CHARACTERIZATION OF NANOFIBER SENSORS

6.3.1 Modulus of Elasticity of Manufactured Nanofiber Sensors

The monotonic tensile tests were conducted for the specimen that had been doped with CNF of weight % of 0, 0.05, 0.08, 0.16, 0.24, 0.4, 0.45, 0.5 and 0.75 and its corresponding E values in GPa were 1.2, 1.8, 1.94, 2.05, 2.69, 2.74, 2.86, 2.94 and 3.4 respectively. During these tests, it was observed that when the wt.% of doped CNF in PVDF fiber mat increased, the corresponding Young’s Modulus also increased. This increment has been sustained till 0.75 wt. % of CNF. This Increase in the wt. % of doping CNF further leads to decrease in the Young’s Modulus. For example, in this study, 1 wt.% of CNF were doped with the PVDF where the Young’s Modulus decreased to 2.8GPa.

Similar procedures were conducted for CNT-PVDF specimen. In this testing, it was identified that the Young’s Modulus of the specimens had gradually increased as the wt.% of CNT is increased up to 0.75%. Further increment of CNT leads to the decrement of the value of Young’s Modulus. As shown Fig. 6.5, from this finding, it could be concluded that the 0.75% shows the better Young’s Modulus for both the fillers such as CNF and CNT. So these specimens were used for further testes like incremental loading-unloading tensile test and 3 point bending test.
6.4 MECHANICAL TENSILE CHARACTERIZATION OF GFRP

6.4.1 Tensile Strength of GFRP without Embedding Sensor

Fig. 6.6 shows the results of tensile strength of GFRP sample without embedding the nanofiber sensors. During this test, the load was increased gradually till the specimen failure occurred, thereby the tensile strength $T$, Strain density $W$, modulus of elasticity $E$ and elongation to fracture $A$ were monitored. The obtained modulus of elasticity for this study is 22.95 GPa, the tensile strength of manufactured GFRP was 506.6 MPa and
the elongation fracture was nearly 2.54%. From this value, the different loading conditions were distributed for the incremental loading and unloading steps.

Fig.6.6 Typical tensile results of GFRP sample without embedding nanofiber

6.4.2 Static Tensile Test to Determine the Change in Resistance at different wt. % of CNF & CNT Nanofiber Mats

The ability of the CNT and CNF with respect to sensing the damage on the composite structures during different loading and fatigue condition need to be analysed. This determines the durability of composite structures and their service life. The damages such as matrix crack, fiber breakage and delamination can be detected by measuring the change in electrical resistance which can be related to the residual strain stored in the
material during loading and unloading condition which could result in damage. It’s necessary to study the behavior of every individual specimen with respect to its change in electrical resistance during mechanical strain and quantifying the sensitivity of the sensor mat as a function to enhance dispersion and surface adhesion.

**Fig. 6.7**: Average normalized change in resistance, $\Delta R/R_0 \times 100$ (%) versus nominal strain (%) for the different weight percentage (wt. %) of CNF fiber mat embedded between the 9th and 10th layer of GFRP composite panel during static tensile test

In the present study, as show in Fig. 6.7 and 6.8, two sets of data have been adopted in which PVDF dispersion with CNF and CNT can be monitored. From this study, the enhanced electrical resistivity during mechanical loading conditions and the weight % of fillers could be
identified. Fig. 6.7 shows the average normalized change in resistance and nominal strain for the different wt. % of CNF such as 0.05wt.%, 0.08 wt.%, 0.16 wt.%, 0.24 wt.% 0.4 wt.% 0.45 wt.%, 0.5 wt.%, 0.75wt.% 1 wt.% of CNF and specimen without embedding CNF fiber mat. When the nominal strain increases, the corresponding change in electrical resistance also increases for all the wt.% of CNF till 0.75 wt.% of CNF, and the change in resistance value also increases. When mechanical loading is applied to the 1 wt. % of CNF, the change in electrical resistance gradually decreased as compared to the 0.75 wt.% . This plot falls between the 0.75 wt % to 0.5 wt. % of CNF. Therefore, the dispersion of the CNF during 1 wt.% is not so homogenous and the solvent quantity need to be improved.

![Graph](image)

**Fig. 6.8** : Average normalized change in resistance, $\Delta R/R_0 \times 100$ (%) versus Nominal strain (%) for the different weight percentage (Wt. %) of CNT fiber mat embedded between the 9th and 10th layer of GFRP composite panel during static tensile test.
The same procedure as CNF has been followed for the CNT samples as well. The results were plotted between Nominal strain [%] verses Average change in resistance [%] for different weight % of CNT fiber mat embedded between the GFRP laminates. Fig. 6.8 presents the results of the Nominal strain [%] verses Average. The average of the five samples has been used for this plot during static tensile test. From the plot, it could be clearly understood that without embedding CNT fiber mat between the GFRP there was no variation in resistance with increasing values of Nominal strain. Hence, the non-conductive GFRP has not shown any variation to the change in resistance during the increase in Nominal strain. When the nominal strain increases, the corresponding change in electrical resistance also increases for all the wt.% of CNT till 0.75 wt.% of CNT, and the change in resistance value also increases. When mechanical loading is applied to the 1 wt. % of CNT, the change in electrical resistance gradually decreases as compared to the 0.75 wt.% . At 1 wt. % of CNT, the change in electrical resistance decreased when compared to the 0.75 wt. % of CNT.

6.4.3 Tensile Strength of GFRP with Embedded CNF/PVDF (0.75wt. %) Sensor at 0° Orientation

Fig.6.9 shows the results of change in resistance during incremental loading and unloading steps in the tensile test. During first loading condition, the nominal stress of 80MPa linearly increased along with the nominal strain. Therefore, the change in resistance during this incremental loading increased slightly. While unloading, the specimen’s response to the change in resistance curve fell on the same pattern as similar as loading condition curve. The next loading steps were carried out and the nominal stress reaches from 114 MPa to 173 MPa with the 2nd and 3rd loading condition and during unloading, the curves followed the same pattern.
Fig. 6.9 Typical tensile mechanical and resistance results of six incremental loading – unloading steps for GFRP samples embedded with 0.75 wt % of PVDF-CNFM nanofiber at 0° orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress.
But 4\textsuperscript{th} and 5\textsuperscript{th} loading and unloading conditions created the hysteresis loop, and while unloading the values, electrical resistance was high when compared with loading case because of this hysteresis. This can be interpreted with damage of the material which means that the electrical resistance value during loading condition is 628kΩ, but the resistance value increased to 641kΩ while unloading. The residual strain measurement was noticed after unloading conditions which means the strain value will not reach zero value during unloading steps and it’s due to material damage.

6.4.4 Tensile Strength of GFRP with Embedded CNF/PVDF (0.75 wt. %) Sensor at 45° orientation

The Fig. 6.10 shows the electrical resistance responses of the CNF nanofiber mat sensor embedded at 45°. The recorded resistance value at 45° orientation specimen was lower than the 0° orientation specimen and the major variation in the diameter of the hysteresis loop at 45° orientated specimens during unloading was less than the previous case and thereby the value during loading and unloading were very close, and the recorded residual strain value was almost half of the residual strain value of the 0° orientations.
Fig. 6.10 Typical tensile mechanical and resistance results of Six incremental loading – unloading steps for GFRP samples embedded with 0.75 Wt% of PVDF-CNf nanofiber at 45° orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress.
6.4.5 Tensile Strength of GFRP with Embedded CNF/PVDF (0.75wt. %) Sensor at 90° Orientation

Fig. 6.11 Typical tensile mechanical and resistance results of Six incremental loading – unloading steps for GFRP samples embedded with 0.75 Wt % of PVDF-CNF nanofiber at 90° orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress
The PVDF-CNFe nanofiber mat is embedded between the GFRP laminates at 90° orientation. Fig. 6.11 shows the electrical resistance response with respect to the nominal strain values. In this plot, the variation in the resistance value was less when compared to 0° orientations. Since this orientation did not give better results even after many testing with different specimen at 90° orientation of fiber mat, another parameter was observed from this study, i.e., it doesn’t create any hysteresis loop. This means the sample follows the same curve for both loading and unloading conditions.

6.4.6 Tensile Strength of GFRP with Embedded CNT/PVDF (0.75wt.% ) Sensor at 0° orientation

Fig. 6.12 presents the typical results of six loading and unloading tensile tests of 0.75Wt % of PVDF-CNT fiber at 0° Orientation. The different incremental loading steps such as 16% of fracture stress, 27% of fracture stress, 39% of fracture stress, 64% of fracture stress, 87% of fracture stress and finally 100% of specimen fracture were involved. It is observed that the change in electrical resistance of the specimen increased with the incremental loading and during unloading condition, the change in resistance value decreased as well.

During 1st three loading conditions, when the applied mechanical load increased, its corresponding strain and change in resistance values increased gradually. Meanwhile, the curve drawn between the strain and change in resistance seemed similar and followed the same pattern for both loading and unloading condition during 1st three loading steps which means there is a decrease in change in resistance value in unloading condition which falls on the same line where the resistance value increases. During the fourth loading and unloading steps, the change in resistance value created a hysteresis loop and simultaneously the change in resistance didn’t return to
zero value during unloading condition which is said to be a residual resistance.

Fig. 6.12 Typical tensile mechanical and resistance results of Six incremental loading – unloading steps for GFRP sample embedded with 0.75 Wt % of PVDF-CNT nanofiber at 0° orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress.
The same sequences such as hysteresis loop and residual resistance have been observed in the next loading and unloading steps. This can be interpreted that the initiation of damage has occurred like fiber matrix crack and debonding in the specimen during the 4th loading which is 64% of the fracture stress. Before 4th loading, specimens gradually increased in nominal strain and change in resistance when the mechanical stress was applied.

6.4.7 Tensile Strength of GFRP Embedded CNT/PVDF (0.75wt. %) Sensor at 45° orientation

Fig. 6.13 shows the results of the tensile test during six incremental loading and unloading conditions of PVDF-CNT fiber mat embedded at 45° orientation. From the plotted curve, it is observed that the nominal strain verses change in resistance plays the vital role to correlate the damage in the materials. In the 1st three loading conditions as mentioned in the previous case that is at 0° orientation, the path of change in resistance during unloading conditions followed the same as loading condition, which means it followed the same pattern for loading and unloading steps. During 4th loading and unloading steps it has been observed that the path of unloading was slightly varied from loading path. Hence, the variation was very small compared to 0° orientation. But even in the 5th loading condition the variation is clearly observed when compared to the 0° orientation. Therefore, it could be concluded that the damage has been initiated from 510Mpa during the tensile loading condition.
Fig. 6.13 Typical tensile mechanical and resistance results of Six incremental loading – unloading steps for GFRP sample embedded with 0.75 Wt % of PVDF-CNT nanofiber at 45° orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress
6.4.8 Tensile Strength of GFRP Embedded with CNT/PVDF (0.75wt. %) Sensor at 90° orientation

Fig. 6.14 Typical tensile mechanical and resistance results of Six incremental loading – unloading steps for GFRP sample embedded with 0.75 Wt % of PVDF-CNT nanofiber at 90° Orientation. 1st loading up to 16% of fracture stress, 2nd loading up to 27% of fracture stress, 3rd loading up to 39% of fracture stress, 4th loading up to 64% of fracture stress, 5th loading up to 87% of fracture stress, 6th loading up to 100% of fracture stress.
As compared to the fiber mat sensor at 0° and 45° orientations, the specimen of GFRP embedded with the CNT-PVDF electrospunned nanofiber mat embedded at 90° orientation seems to possess poor response to change in electrical resistance during mechanical loading and unloading conditions. As stated in section 6.4.7 and shown in Fig. 6.14, the nominal strain doesn’t create any hysteresis loop during loading and unloading condition which means there is no residual strain stored in the specimen. The change in resistance and nominal strain follows same path during loading and unloading steps. Hence, fiber mat sensor at 90° orientation of nanofiber mat, the specimen doesn’t respond to the change in electrical resistance as in the sensor with 0° and 45° orientation. Thereby from this study it’s clearly observed that the 0° orientation which is axial to the tensile loading sensor gives the better response to the change in electrical resistance during loading and unloading condition and hence damage interpreted.

6.5 DIRECT CORRELATION BETWEEN MECHANICAL STRAIN (VS) ELECTRICAL RESISTANCE FOR 0° OPTIMIZED ORIENTATION OF EMBEDDED PVDF-CNF & CNT SENSORS

In order to correlate the gross mechanical stress and $\Delta R/R_0$ values, the nominal stress instead of nominal strain should be in x axis. Therefore, the mechanical strain is linearly related with modulus of elasticity ‘E’. The mechanical stress is highly preferable for aerospace design parameter due to its mechanical property. From the previous section, it could be observed that the optimized orientation of the embedded sensor for the tensile loading and unloading conditions is with fiber mat sensor at 0° orientation in the loading direction. In that orientation, the electrical resistance response of the nanofiber mat was better during loading and unloading steps. During correction all the plots were matched with the exponential growth curve of equation 6.1.
\[ y = A \cdot e^{x/t} \] (6.1)

In Table 6.1 and 6.2, the calculation of A and t for the exponential growth curve of various wt% of CNF-PVDF specimen and CNT-PVDF specimen with different orientations are shown respectively, and Fig. 6.15 and 6.16 show the graphical representation of the test results respectively.

Fig.6.15 Typical results of direct correlation between the mechanical stress and ratio between the change in resistance at 0° orientation of PVDF – CNF nanofiber sensor embedded between the GFRP laminates at different loading conditions
Fig. 6.16 Typical results of direct correlation between the mechanical stress and the change in resistance at 0° orientation of PVDF–CNT nanofiber sensor embedded between the GFRP laminates at different loading conditions like. (a) 1st loading (b) 2nd loading (c) 3rd loading (d) 4th loading (e) 5th loading (f) 6th loading

In CNF-PVDF sample, the mechanical stress of up to 259MPa shows the change in electrical resistance measurement curve following the
same exponential curve. Once the applied load is increased, the change in resistance also increases to very high values which alter the applied mechanical load that exceeds the safe zone. Therefore, the material has been initiated to damage. Usually there might be matrix cracking (or) debonding of reinforced fiber. In CNT-PVDF sample, when the mechanical load applied up to 400MPa with respect to the change in electrical resistance the exponential growth curve follows the same exponential curve. When the applied mechanical stress has been increased further from 4th loading and 5th loading, the damage starts when the exponential growth curve shifts from its path and this can be interpreted in two ways - either there is some damage pertained in CNT fiber or in the reinforced fiber. Hence, there is no possibility of nanofiber damage, however, it can withstand up to 200% of mechanical strain of the CNT fiber (Alexopoulos, et al., 2008), but, according to this reference, the strain value obtained here is in the order of 1-2%. Therefore, there may be possibility of reinforced fiber damage in terms of matrix cracking (or) debonding.

6.6 ELECTRICAL RESISTANCE CORRELATING TO THE DAMAGE BY PROGRESSIVE DAMAGE ACCUMULATION TEST (PDAT)

In both PVDF-CN and PVDF-CNT fiber sensor mats, during 1st four loading and unloading conditions, the change in electrical resistance follows the same curve for loading and unloading steps. The electrical resistance measurements for the materials remain the same (628kΩ). But after 4th unloading step, the electrical resistances gradually increased around 641 kΩ. Therefore, the unloading curve does not follow the same curve of loading steps which shows the possibility of material (or) fiber damage. Consequently, the hysteresis loops were noticed for both 4th and 5th unloading which, after 400MPa of mechanical stress, which indicates the damage initiation to damage.
Table 6.1 Direct correlations between the mechanical stress, electrical resistance and normalized modulus of elasticity by exponential growth curve equation during loading and unloading conditions for the PVDF-CNF nano fiber sensor mat embedded between the GFRP samples

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<th>Loading steps</th>
<th>Percentage fracture stress during loading conditions [%]</th>
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<th>t[-]</th>
<th>E/E₀ [%]</th>
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Table 6.2  Direct correlations between the mechanical stress, electrical resistance and normalized modulus of elasticity by exponential growth curve equation during loading and unloading conditions for the PVDF-CNT nano fiber sensor mat embedded between the GFRP samples

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<th>Percentage Fracture Stress During Loading Conditions [%]</th>
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<th>t[-]</th>
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Fig. 6.17 Results of progressive damage accumulation test were the degradation in modulus of elasticity of all the samples such as with and without embedding PVDF-CNF nanofiber sensor mats, GFRP embedded with ultrasonic sensor and piezoelectric sensor.

The GFRP specimens used for progressive damage accumulation test are without and with embedding CNF-PVDF nanofiber at different wt. %, embedded with ultrasonic and piezoelectric sensors. During six loading and unloading steps, as shown in Fig. 6.17, their corresponding nominalized modulus of elasticity with respect to the fracture stress were monitored and plotted, and Fig. 6.18 shows the effects of modulus of elasticity when CNT-PVDF nanofiber embedded between the glass fiber laminates.
Fig. 6.18 Results of progressive damage accumulation test were the degradation in modulus of elasticity of all the samples such as with and without embedding PVDF-CNT nanofiber sensor mats, GFRP embedded with ultrasonic sensor and piezoelectric sensor. Since the test was load controlled, at last unloading conditions the specimen returns to zero load condition. The artificial damage was introduced to the specimen through adding loads of 50MPa and this damage was noticed as the degradation of material stiffness. And the same procedures were followed in CNT specimen. Quantating the damage mechanism in the composite structure can be done in many ways such as ultrasonic sensors (Pantelakis et al. 2001), AE technique (Loutas, et al. 2009) and acousto-
ultrasonic (Philippidis, et al. 2008). But in the mechanical loading and unloading point of view, the damage can be quantified by the value of modulus of elasticity (E/E<sub>0</sub>).

Based on the magnitude of the load different types of damage can develop in the composite structures. If the peak load value is low there may be a matrix cracking and debonding occurs between the matrix and fiber. If the peak load value is medium there may be the possibility of delamination on the fiber matrix and if the loading value were very closed to the ultimate tensile load, there may be fiber breakage. The development of the damage and its location mainly depend on the damage mechanism of the composite, which can be interpreted by increasing the loading steps. From the observation the 0.75Wt.% of nanofiber mat for both CNT and CNF gives the better modulus of elasticity when compared with others. The embedded sensor of piezoelectric and ultrasonic sensor shows the lower modulus of elasticity when compared with the nanofiber embedded sensors.

### 6.7 THREE POINT BENDING TEST

Three point bending tests were conducted in 2 ways for embedded PVDF-CN and PVDF-CNT fiber mat. In this test 0.75 wt.% of CNF and CNT specimen were considered since the modulus of elasticity for this sample is better when compared with other specimens.

The two different types of tests are:

(a) The embedded PVDF-CNT/PVDF-CN fiber mat placed at the bottom of the specimen where the embedded fiber experienced tensile stress.
(b) The embedded PVDF-CNT/PVDF-CNF fiber mat placed at the top of the specimen where the embedded fiber experienced compression stress

6.7.1 CNF/PVDF Nanofiber in the Tensile Region

During this test the embedded fiber mat sensor in the GFRP composite structure was placed at the bottom of the specimen where the tensile stress has developed in the region of the fiber.

6.7.1.1 Monotonic fracture test at 0° orientation

Fig. 6.19 shows the results of the mechanical stress/strain as well as ratio of $\Delta R/R_0$ measurement of monotonic test until the specimen fractured. During Monotonic test, the mechanical load is converted to the mechanical stress and this load is applied till the specimen fractured and its corresponding strain and change in electrical resistance were analyzed by exponential curve fit.

In this case, the linear curve fit resulted in poor correlation where $R^2 = 0.89$, but the better exponential curve fit will be at $R^2 = 0.98$. So, the specimen was tested by various incremental loading and unloading steps in order to determine the exact loading step where the specimen were initiated to damage.
Fig. 6.19 Typical 3 point bending test results were PVDF-CNCF nanofiber mat under tension for a monotonic loading till the specimen fracture

6.7.1.2 Incremental loading and unloading at 0° orientation

From the monotonic test, the maximum mechanical stress was determined, from this values the incremental loading steps were distributed in range of 16% of fracture stress as the 1st loading condition; 38% of fracture stress as the 2nd loading condition; 50% of fracture stress as the 3rd loading condition; 68% of fracture stress as the 4th loading condition; 87% of fracture stress as the 5th loading condition and 6th loading till the specimen fracture. Figs. 6.20 to 6.25 show these variations in the conditions.

When the mechanical stress increases in the 1st loading step, the corresponding strain value and change in electrical resistance increases gradually and during unloading the electrical resistance decreases as the strain value decreases and both the curves follow the same pattern. The values are small and noisy.
Fig. 6.20 Typical results of 1st incremental loading and unloading steps in 3 point bending test were the PVDF-CNFM fiber mat under tension

Fig. 6.21 Typical results of 2nd incremental loading and unloading steps in 3 point bending test were the PVDF-CNFM fiber mat under tension
Fig. 6.22 Typical results of 3\textsuperscript{rd} incremental loading and unloading steps in 3 point bending test were the PVDF-CN\textsuperscript{f} fiber mat under tension

Fig. 6.23 Typical results of 4\textsuperscript{th} incremental loading and unloading steps in 3 point bending test were the PVDF-CN\textsuperscript{f} fiber mat under tension
**Fig. 6.24** Typical results of 5th incremental loading and unloading steps in 3 point bending test were the PVDF-CN fiber mat under tension

**Fig. 6.25** Typical results of 6th incremental loading and unloading steps in 3 point bending test were the PVDF-CN fiber mat under tension
The third loading and unloading was conducted where the mechanical stress is 200MPa which was 50% of fracture stress. While unloading, the embedded fiber response was very distinctive and they created the hysteresis loop. This hysteresis loop was created due to the $\Delta R/R_0$ measurement during unloading. When the mechanical stress comes to zero, the change in resistance didn’t return to zero where the residual electrical resistance was created. In the next loading step, a residual electrical resistance of approximately 8kΩ resulted, which can be interpreted that after 3rd loading condition the material damage has initiated. In the last loading condition, till the specimen fracture the change in resistance increased.

6.7.2 CNF/PVDF Nanofiber in the Compression Region

During this test the embedded fiber mat sensor in the GFRP composite structure was placed on the top of the specimen where the compressive stress has developed in the region of the fiber.

6.7.2.1 Monotonic fracture test at 0° orientation

In monotonic three point bending test, the mechanical load was applied till the specimen fractured, from this test the maximum stress can be obtained. During this test, the embedded fiber’s change in electrical response and strain values are monitored. When the mechanical loading increases continuously, the PVDF-CN fiber’s electrical resistance response is in the negative direction. This is clearly shown in Fig. 6.26. At the lower strain values the change in electrical resistance remain in negative direction and when the mechanical load further increases the corresponding change in resistance move slowly to the positive peak where the embedded fiber experiences the tensile region.
Fig. 6.26 Typical 3 Point bending test results were PVDF-CNF nanofiber mat under compression for a monotonic loading till the specimen fracture

6.7.2.2 Incremental loading and unloading at 0° orientation

From the monotonic test the maximum stress value were determined for the GFRP embedded with PVDF-CNF nano fiber mat under compression loading till the specimen fracture. From the obtained maximum stress value, the incremental loading steps were calculated as 16%, 38%, 50%, 68%, 87% and 100% till fracture for 1st, 2nd, 3rd, 4th, 5th and 6th loading conditions respectively.
Fig. 6.27 Typical results of 1st incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNF fiber mat under compression.

During the 1st loading condition, when the mechanical stress increases and the corresponding strain value also increases, the change in electrical resistance moves towards negative direction. This movement is shown in Fig. 6.27, and this continued to the 2nd loading condition as plotted in Fig. 6.28. A region where the negative peak was noticed for the change in electrical resistance measurement which is approximately 0.72% stain value and its corresponding mechanical stress is 50MPa, known as the compressive region.
Fig. 6.28  Typical results of 2nd incremental loading and unloading steps in 3 Point Bending test were the PVDF-CN fiber mat under compression

Fig. 6.29  Typical results of 3rd incremental loading and unloading steps in 3 Point Bending test were the PVDF-CN fiber mat under compression
Fig. 6.30 Typical results of 4th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNf fiber mat under compression.

Fig. 6.31 Typical results of 5th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNf fiber mat under compression.
Fig. 6.32  Typical results of 6th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNf fiber mat under compression

After 3rd loading condition, the change in electrical resistance values move towards positive peak as shown in Fig. 6.29, this region is where the embedded fiber mat experiences the tensile load. And in additional to this from the 1st loading to the 5th loading condition, during unloading the change in electrical resistance does not return to zero. As shown in Fig. 6.30, 6.31 and 6.32, some of the residual resistance were retained in the materials, and this sign gives the alter signal that the materials were initiated to damage.

The embedded fiber’s electrical resistance response were recorded both in positive and negative values, where the fiber’s region is in tension and compression respectively. In addition it gives the proof that at each loading condition the embedded nanofiber sensor mat was working. But in the compression loading condition when the fiber was placed in the top layer of GFRP laminate, the material got damaged during 1st loading step.
6.7.3 CNT/PVDF Nanofiber in the Tensile Region

6.7.3.1 Monotonic fracture test at 0° orientation

Fig. 6.33 Typical 3 Point bending test results were PVDF-CNT nanofiber mat under tension for a monotonic loading till the specimen fracture

Fig. 6.33 shows the mechanical and electrical behaviours of the CNT fiber at 0° orientation during three point bending test under monotonic loading conditions till the specimen fracture. The direct correlation between the mechanical stress and electrical resistance was analyzed using 3 Point bending test.
6.7.3.2 Incremental loading and unloading at 0° orientation

From the monotonic 3 point bending test, the maximum fracture stress of the specimen was determined. The fiber’s response in terms of electrical resistance and mechanical stress was analyzed in another specimen under six loading and unloading conditions. Figs. 6.34, 6.35, 6.36, 6.37, 6.38 and 6.39 show the six loading steps are 18%, 38%, 50%, 67%, 83% and 100% of the fracture stress and it’s corresponding plots respectively.

![Graph of incremental loading and unloading](image)

**Fig.6.34** Typical results of 1st incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension

For the first two loading conditions till 38% of fracture stress, the change in electrical resistance response was very small and noisy values were monitored and during the third loading condition two parameters were observed, (a) the hysteresis loop during loading and unloading condition, and
(b) the change in resistance will not return to zero value where the residual resistance was observed.

![Fig.6.35 Typical results of 2nd incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension](image-url)
Fig. 6.36 Typical results of 3rd incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension.

Fig. 6.37 Typical results of 4th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension.
Fig.6.38  Typical results of 5th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension

At the 5th loading condition which is 83% of the fracture stress, the specimen has undergone damage which is clearly understood from the residual resistance ratio. Till the 5th loading–unloading condition, the change in resistance ratio is in the order of 0.01%, approximately 6kΩ, which is $\Delta R/R_0 = 0.01$, but during the 5th loading condition the change in electrical resistance value increases drastically around 0.08 and it increased further till the fracture of the specimen. When correlating this value with the mechanical stress and change in resistance, it is directly quantified same as the result of tensile test. When the damage occurs to the materials the curve correlation between the mechanical stress and $\Delta R/R_0$ has shifted from the original position and it becomes the function of damage in the material.
Fig. 6.39 Typical results of 6th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under tension
6.7.4 CNT/PVDF nanofiber in the Compression Region

6.7.4.1 Monotonic fracture test at 0° orientation

When the monotonic 3 point bending test was conducted till the fracture of the specimen, its mechanical stress-strain and its change in electrical resistance were monitored and plotted in Fig. 6.40. From the curve it is clearly observed that, when the mechanical loading increased continuously, the embedded CNT fiber’s electrical resistance response reads towards negative. In the compression region, the negative peak was noticed at 0.64% of nominal mechanical strain and its corresponding mechanical stress was almost 62Mpa. Hence, the negative peak is the sign of alert, that the material was damaged. This is due to the large deflection of specimen in it cross sectional area during loading condition. After that the mechanical loading was increased further and its change in electrical resistance response increased with the strain value which concludes that the fiber was in tension...
region in higher strain values. And the mechanical stress value is further increased till the specimen fractured; still the embedded fiber was in tension region.

6.7.4.2 Incremental loading and unloading at 0° orientation

![Graph showing nominal stress vs. nominal strain for incremental loading and unloading steps.](image)

**Fig. 6.41** Typical results of 1st incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression

Fig. 6.41 to Fig. 6.46 show the results of mechanical stress and its corresponding change in electrical resistance and nominal strain when 3 point incremental loading and unloading steps. During the test the embedded PVDF-CNT fiber mat was embedded between the 9th and 10th layer of glass fiber laminates and it was placed in the top of the specimen where compressive stress will be experienced by the fiber mats.
Fig. 6.42 Typical results of 2\textsuperscript{nd} incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression.

Fig. 6.43 Typical results of 3\textsuperscript{rd} incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression.
Fig. 6.44 Typical results of 4\textsuperscript{th} incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression

Fig. 6.45 Typical results of 5\textsuperscript{th} incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression
Fig. 6.46 Typical results of 6th incremental loading and unloading steps in 3 Point Bending test were the PVDF-CNT fiber mat under compression

During 1st two loading and unloading conditions the change in electrical resistance response of the embedded PVDF-CNT fiber mat’s flows towards negative direction which is as similar as monotonic results during low strain values and another observation was that there is no residual resistance stored during unloading conditions.

But after 3rd, 4th and 5th loading conditions where the strain value is higher as compared with first two loading step and its corresponding electrical resistance increases thereby it is concluded that the embedded nano fiber mat was in tension region. When the strain value is high its electrical resistance increases and during unloading the electrical resistance creates the hysteresis loop, the embedded fiber mat measures the residual resistance during unloading condition which is due to the damage accumulation. The embedded PVDF-CNT fiber recorded both negative and positive peak which may In the next loading step, a residual resistance of approximately 8kΩ resulted, which
can be interpreted that after 3rd loading condition the damage in the material has initiated.

6.8 CONCLUSION

In the surface morphology study, the electrospun PVDF doped with CNF and CNT were examined. Moreover, their smooth surface, individual nanotube, uniform surface morphology were visible at 0.75 wt%, and the beads were observed in 1 wt. % doped fillers fiber mat due to instability of the jet flow and non-homogeneous mixture. As far as this study is concerned, it indicates that 0.75wt % of PVDF fiber mat sensor doped with CNF and CNT are the optimized sensor for structural health monitoring due to the lack of agglomeration and presence of good dispersion.

In crystalline morphology, the peak was obtained at 20.7 (200) at 0.75 wt. % which shows the presence of β phase and this implies the significant strength of increasing piezoelectric conductivity in both CNF and CNT doped fiber mats.

The Young’s modulus of the specimen was analyzed, and the analysis showed that the specimen with 0.75 wt. % of CNT and CNF doped fiber mat has enhanced modulus of elasticity. From these tests, it has been concluded that 0.75 wt. % of CNF and CNT doped specimens have good dispersion, enhanced β Phase and improved Young’s Modulus which has been used for further mechanical characterization.

The static tensile test was conducted to determine the average change in resistance for different wt. % of CNF and CNT samples. Since the CNF and CNT are electroactive material that which generates a signal in response to a mechanical force. Out of these, 0.75% wt. shows the better change in resistance during the increment of nominal strain.
The specimen with 1% wt. shows a decrease in young’s modulus value which means interaction between the fillers and PVDF matrix, is weak and there are no agglomerates of MWCNT which could act as a mechanical interlocking between polymer chains and the fillers. This is evident also from the slight decrease of yield strain and maximum strain at 1% wt. filler content. At 0.75% wt. of filler, the yield stress has slightly increased which has the attribute to enhance the β-phase and could be responsible for the small increase in hardness and strength of PVDF/CNT and PVDF/CNF composites. The β-phase decreases the plastic property in the materials under applied pressure. This may lead to retarding the formation of cracks.

Tensile test was carried out in 2 ways – (i) Monotonic tensile test and (ii) incremental loading and unloading test. In the monotonic test, the specimens were loaded till they fractured and from that the maximum fracture stress were determined. These stress values were distributed into 6 incremental loading steps. During each loading step, the fiber mat sensor’s response was monitored in terms of change in electrical resistance which directly correlated with structural damage at the different orientation of fiber mat sensors like 0°, 45° and 90°. Among these orientations, 0° orientation gives the better results and it’s suitable for damage sensing in the aircraft structures. The 3 point bending tests were conducted in 2 types namely compression and tension bending. The embedded fiber mat sensor experiences the compression and tension at 0° orientation. In the compression region, a negative peak was noticed at 0.64% of nominal strain, this is due to the large deflection of the specimen in its cross sectional area during loading condition.