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TEMPERATURE SENSING SELF ACTUATING POLYMER NANO-COMPOSITES SURFACE MORPHOLOGY STUDY

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ABSTRACT

Research work reported herein, involve fabrication of multiwalled carbon nanotubes (MWCNTs) (MWCNTs type 3, MWCNTs type 5 and MWCNTs type 6), reinforced into shape memory thermoplastic polyurethane (SMTPU) by use of melt mixings route by the use of micro-compounder followed by mini injection moulding. The composites of multiwalled carbon nanotubes (MWCNTs) were prepaired through ex-situ polymerization, into SMTPU pure matrix, containing 0 to 1.5 phr (par per hundred) fillers. Successfully dispersion was achieved for various percentage of nano fillers into SMTPU pure matrix which is characterized using Fields Emissions Scanning Electrons Microscope (FE-SEM), High Resolution Transmission Electron Microscope (HR-TEM), Atomic Force Microscope (AFM), and X-Rays Diffractions (XRD) etc. the shape and size of nano fillers (MWCNTs and graphene nanoplatelets) was characterized using high quality TEM and FE-SEM, and AFM techniques.

Keywords: Shape Memory Materials, Polymers, Smart Actuators.

I. INTRODUCTION

The mechanism behind the SMP consists of two-phase, one is the hard phase and another is soft phase. Soft phase is an elastic phase which is responsible for switching temporary shape. And hard phase is fixing phase which is responsible for regaining original shape [1-2]. The shape is changing temporarily to permanent below and above the glass transition temperature (Tg). So that glass transition temperature plays an important role in shape memory polymers. Below the glass transition temperature polymer behave like a glass so it's called as glassy state and above glass transition temperature it behaves like a rubber so it's called rubbery state. SMPs generally used for sensors and actuators devices for various sophisticated applications but SMPs Frequently used in aerospace, industrial, medical, automobiles, various research and developments etc. in general, shape memory polymers are triggered by the conventional method by means of heat, water, electric, magnetic, ph, solution, electromagnetic etc. but it's usually impossible to trig the SMPs by conventional method during operating time especially for space and remote sensing devices [3-6]. So that for sophisticated wireless applications, nonconventional method comes into the picture. Nanomaterials are most popular reinforced materials for shape memorized polymer due to their excellent thermal, electrical properties and mechanical properties. Several scientific studies reported that [4, 7-10], the improvement in properties of the shape memory on the addition of nanofillers which includes reinforcement of nanoclay, SiC, carbon black, CNT, cellulose nanowhiskers, graphene etc. Among such usual used fillers, carbon-based filler is popular which include mMWCNTs, carbon black, carbon nanopaper and graphene.

Amongst various fillers, CNTs and carbon black are extensively studied to improve mechanical and electrical properties. Research shows that the filler geometry, method of mixing and concentration are akey factor governing composite properties. SMP/Graphene nanocomposites drew the researcher's attention due to their one sided unique electrical properties and mechanical property. Graphene is a one-atom-thick two-dimensional sheet of graphite having sp2 hybridized carbons is covalently bonded in a hexagonal manner. Its high stiffness, high aspect ratio, superior electric and thermal conductivity makes graphene a suitable candidate as reinforcement in the polymer matrix. Graphene nanoplatelets (GNP) are newly developed the short bulk form of graphene atoms. Many researchers investigated triple way photo-responsive shape memory polymer/azobenzene and graphene oxide (GO) nanocomposites film were prepared. The azobenzene and GO were acted as a heat source for heating the polymer film, which is responsible for shape recovery. Mechanical and shape memorized properties was improved in UV and NIR light-responsive nanocomposites. Optical responsive shape memory polymer chain for write and erase fingerprint pattern was reported. Photo-responsive shape memory polymer composites exhibit low cost, ease of available and fast shape recovery for sensors and actuators. Photo-responsive SMP graphene oxide composites were prepared by solvent casting

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route for photo-mechanical applications. In photo-mechanical polymer/GO nanocomposites graphene oxide is responsible for heat transfer in the polymer matrix. Graphene is superior thermal, mechanical and electrical properties which are used in sensors and actuators devices [11-13].

Shape memorized stretch and recovery tests were performed by using the tensile testing with wedge clamps gripped machine. In the shape memory stretch test the sample size, 50x10x1 mm^3 was clamped in grips of springs loaded with wedge shape and heated at 74°C to become completely rubbery state. At 74°C, the force is applied at the rate of 2.5 mm/minutes upto 100% strain. Thereafter, furnace chamber is open to cool the sample below 37°C (room temperature) without releasing the load. Once the sample cools below 37°C the sample removes from the grip and measure the final gauge length for shape fixity calculation. And after measuring the shape fixity the stretched sample again clamp in the grip for measuring constrained shape stretch recovery force. For a constrained shape recovery stress tests, the stretched sample clamp in grip and heating the sample upto 68°C to measure the recovery force. During the recovery test the recovery force initially drastically decreased, after that it goes to constant after some time.

UV and NRI light responsive shape memory polymer composites reinforced with CNT were investigated by researchers for fingerprint pattern write and erase applications. Our previous research also reported that MV-induced polyurethane/GNPs nano-compositeswere successfully trigged and properties were significantally improved with the reinforcement of GNPs in the PU matrix. Our previous research for improving mechanical and shape memory properties the (GNPs) grapheme nanoplatelets were used in polyurethane matrix. Shape memory polymer composites reinforced with CNTs/graphene strongly influence by moisture, the concentration of reinforcements and distance of dispersed reinforcements within polymer matrix. Microwave induced shape memory polymer composites components are volumetrically heated which may help for uniform and fast actuating applications [1-5, 24].

II. RESULTS & DISCUSSIONS

Samples of pure SMTPU and composite contains 0 to 1.5 phr (part per hundred) and 0 to 1.5 phr MWCNTs was prepare by the melt mixings routes in the Thermal Haeke MiniLaboratory 3 (PolyLabs model OSo) macro-compounders holding twin conic angle screws followed by injections mini mold Thermal Scientifics German (HAEKE Mini-jet Pistons Pro) were used for samples preparations. Firstly SMTPU, and MWCNTs was dried into the vacuumed ovens for the time 2 hours at 100°C to removes the carbonaceous and moisture impurities. After that, a weighted amounts of the SMTPUs granule and or MWCNT powder (anyone at a time) was proceed in macro-compounders followed by mini injections molding. During the samples making, 5g SMTPUs granules and weighted amounts of phr MWCNT have take and process into macro-compounder; speed of twins conic screws were 60 rpm, temperature of mixing is 210°C for the time 10 minute. Then after melts mixture were taken into cylinder which are preheated heated at 200°C and processed for Injections mini Mould. Different compositions of PU with 0, 0.5, 1and 1.5 phr GNPs or MWCNTs was prepare in batche of 5g each into macro-compounder. To obtain uniform composition each 5g batche of the mixtures were extrude in the form of the wires then chopped and chopped was reprocessed in micro compounder.

The shape memory thermoplastic polyurethane (SMTPU) and composites of multiwalled carbon nanotubes (MWCNT type 5) and graphene nanoplatelets (GNPs) containing 0 to 1.5 phr (part per hundred) were prepared in micro-compounder followed by injection moulding through ex-situ polymerization technique by using melt mixing route. The work reported herein, composites of MWCNTs and GNPs both were prepared by using 0, 0.5, 1 and 1.5 phr concentrations into neat SMTPU base matrixThe melt mixture from micro-compounder is directly made by injection moulded in standared (ISI) shape and size (length of gauges, thickness, and width for the sample was kept 18.5, 3 mm, and 5 nm respectively, for tensile test and for Dynamic Mechanical Analyzer tests the size of samples are 40X10X1 mm^3) at temperature of moulding 190°C, pressure of injection moulding 500bar, injection rate 6 seconds and temperature of mould 85°C. After that 5-8 minutes, the samples removed from the die (ISI) for characterizations and testing of samples.

In present experiments the materials used is brought from, Graphene nanoplatelets (GNPs) having 11-15 nm in powder form (SMTPU) Thermoplastic shape memory polyurethane ether type granules (MM 6520) purchased from SMP Intec. Diplex (Mitsubishi), Japan. Multiwalled carbon nanotubes (MWCNT) containing's, different

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three types, variable same length and outer diameter's (Type 6:OD= > 50 nm, L= 10-30 um Type 3: L= 10-30 um , OD= 10-20 nm and, Type 5: L= 10-30 um, OD= 30-50 nm) are purchase through Siscos Researchs Laboratorie Pvt. Ltd. Supply through Nano Shels Pvt. Ltd. is purchases from Siscos Laboratorie Researchs Pvt. Ltd.

The shape memory thermoplastic polyurethane (SMTPU) and composites of multiwalled carbon nanotubes (MWCNT type 5) and graphene nanoplatelets (GNPs) containing 0 to 1.5 phr (part per hundred) were prepared in micro-compounder followed by injection moulding through ex-situ polymerization technique by using melt mixing route. The work reported herein, composites of MWCNTs and GNPs both were prepared by using 0, 0.5, 1 and 1.5 phr concentrations into neat SMTPU base matrix. Further, the composite samples containing 1.5 phr Multiwalled carbon nanotubes (MWCNT) containing's, different three types, variable same length and outer diameter's (Type 6:OD= > 50 nm, L= 10-30 um Type 3: L= 10-30 um , OD= 10-20 nm and, Type 5: L= 10-30 um, OD= 30-50 nm) was also prepared by using same micro-compounder through melt mixing route. The detailed composition and designation as well as processing parameters were explained in materials and experimental section. Dispersion of targeted percentage (0 to 1.5 phr) of nano fillers (MWCNTs/GNPs) into SMTPU matrix which is characterized by using Field Emissions Scanning's Electrons Microscope (FE-SEM), Atomics Force Microscope (AFM), High Resolutions Transmissions Electrons Microscopy (HR-TEM), and X-Rays Diffractions (XRDs) technique. The shape and size of nano fillers (MWCNTs and graphene nanoplatelets) were characterized by using high resolution TEM, FE-SEM, and AFM techniques. Microwave induced thermal shape recovery tests for pure SMTPU and composites sample were conducted using microwave oven. Simultaneous microwave induced shape recovery phenomenon was recorded by the use of excellent quality thermal imager of infrared. Transition temperature of glass was calculated by using DSC and DMA curves for pure and composite samples.

Mechanical property and shape memorized property were improve with reinforce of reinforced MWCNT/GNPs filled into pure SMTPU matrix. Filler materials and soft segments of SMTPU polymer make strong interfacial interaction during sample preparation. Soft segments and filler also make physical cross-linking and restrict the chain movements of polymer base matrix results improved shape memory as well as mechanical property. Maximum shape memorized and mechanical properties were observed for composites having 1.5 phr fillers. Furthermore, the 1.5 phr GPU composites exhibited better properties as compared to 1.5 CNTPU composites. For moisture induced samples, molecules of water react with soft segments of polyurethane and make hydrogen bond in-between C=O and H-O group and chain of polymer may get weak. Shape memory and mechanical properties were suppressed for moisture induced sample but the microwave induced shape recovery is faster as compared to without water immersed samples. The shape recovery for 10 days water immersed 1.5 GPU and 1.5 CNTPU composites sample is much faster and shows approximately 100% shape recovery. Microwave induced and moisture shape recovery stimuli may open research gate for non-contact heating, wirelessly, clean green source of heating as well as fast and efficient heating.

2.1 Field Emissions-Scanning Electrons Microscope (FE-SEM): Cryogenics morphological fractured surface were analyzed by the use of (FE-SEM of M/s 430NanoSEM). The (LN) liquid-nitrogen was used for the cryogenic fracture samples and fractured samples were gold coated at 100 Å for clear observations of surface morphology.

FE-SEM characterization was used to observe the distributions of nano fillers and surface morphology of pure and composites samples. Fig. 5.1 shows the surfaces morphology of cryogenics fracture of (a) pure SMTPU, (b) of MWCNT type 5, (c) of 0.5 CNTPU, (d) of 1 CNTPU, and (e) of 1.5 CNTPU composite samples. For pure SMTPU (Fig. 5.1(a)) smoother, uniform pattern, and layered fracture surface was observed. Fig. 1(b) shows the dimensions of used MWCNTs type 5 (L= 10-30 um, O.D= 30-50 nm). The roughness of fractured surface increases due the additions of MWCNTs into polyurethane matrix. For composite samples the random and proper distributions of MWCNTs into SMTPU matrix were shown clearly (Fig. (c-e)). MWCNTs and SMTPU makes strong interfacial interaction due to adhesion between them. Adhesion between MWCNTs and polymer matrix restricts the chain movements of soft segments of polymer matrix results in blocked crack propagation during fracture and properties of composites may enhance.

In Fig 5.1 fractured surface morphology of PU, this shows that smooth, layered and uniform orientation. Random and homogeneous distribution of nanoplatelets helps to superior properties. MWCNTs and GNPs fillers makes physical cross linking with soft segments of SMTPU matrix and the movements of chain get

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restricted. In Fig. 5.2 (d) microcracks and agglomeration also observed in some places which are responsible for the inferior shape memorized as well as mechanical property.

2.2 Transmission Electrons Microscope (TEM): Transmission Electrons Microscopy is the excellent resolution surface characterization as well as surface morphology characterization technique in which electron bean is transmitted through the targeted sample. The targeted sample should be very thin less than 100 nm or is suspended into a grid. The samples of pure MWCNTs figure 5.1 MWCNT composite SEM image, were suspended into grid for characterization of size and shape. Fig. 5.2 shows that the TEM images of pure MWCNTs type 5. Fig. 5.3(a-c) TEM image of graphene nanoplatelets powder having higher resolutions and Fig. 5.4 (d-f) image of MWCNTs type 5 having higher resolutions for clearer observations of shape and size of nano fillers.

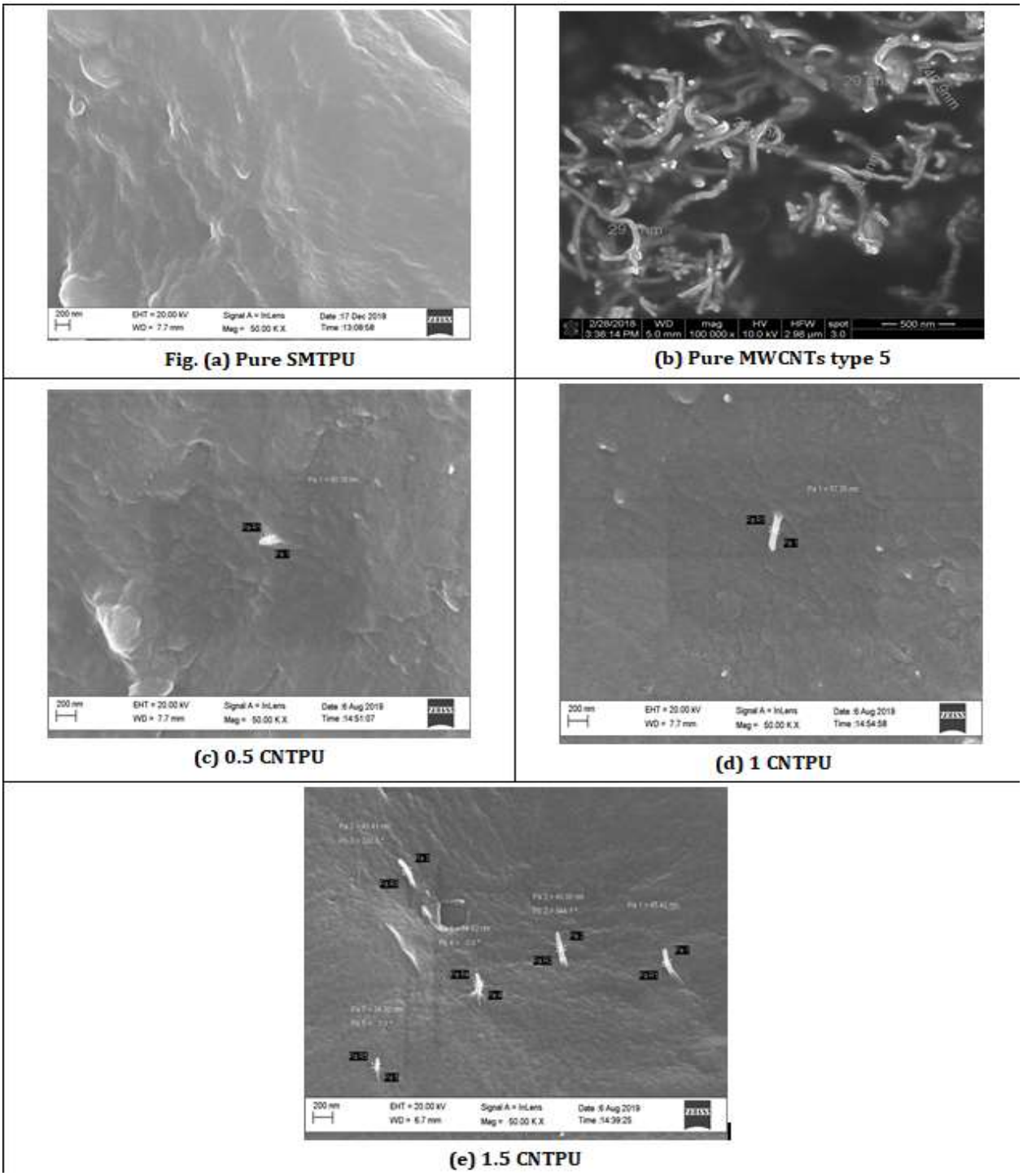

Fig 2.1: FE-SEM Cryogenics fracture surfaces morphology of the pure, MWCNTs and composites samples.

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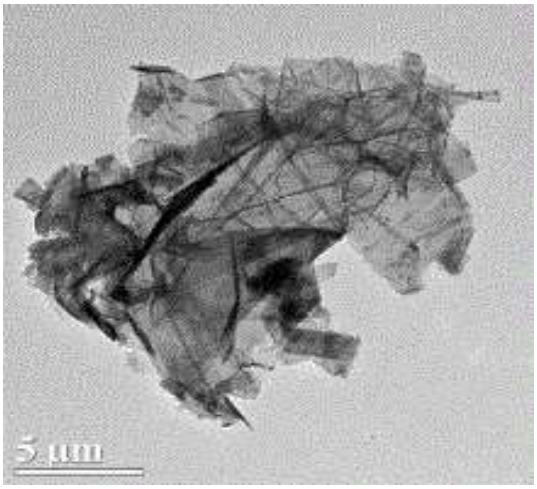
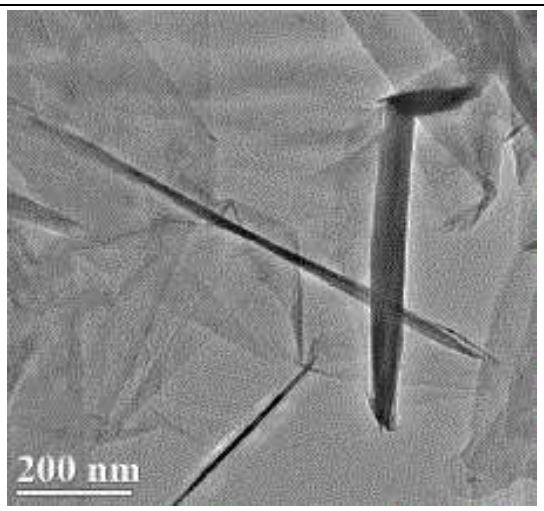
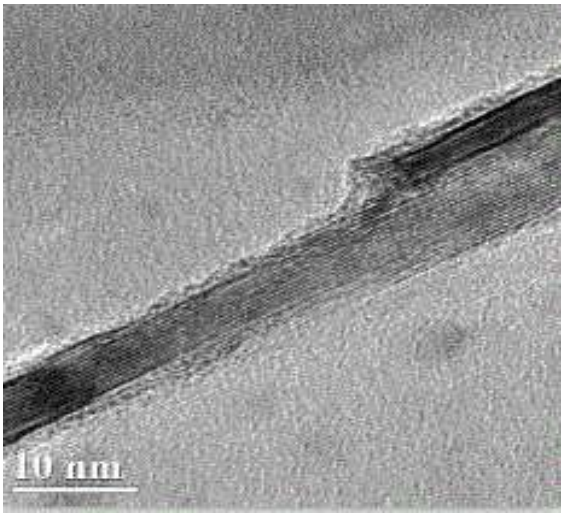


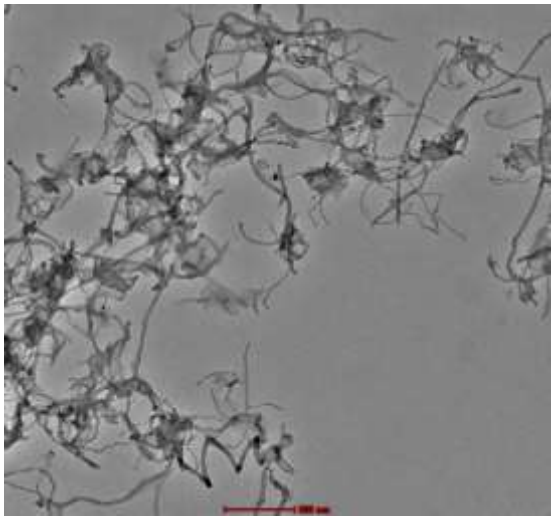
(a) Graphene nanoplatelets



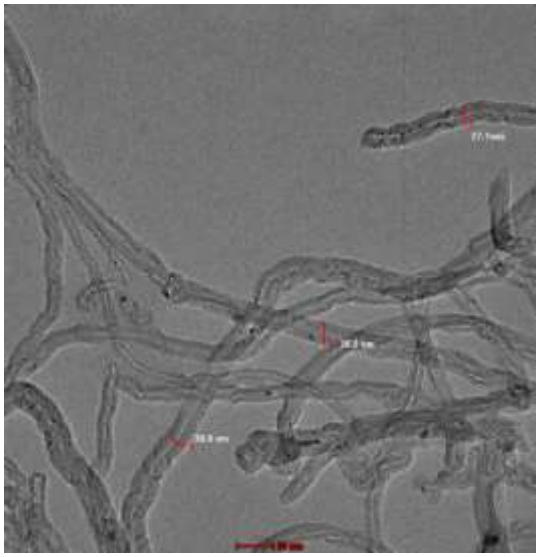
(b) Graphene nanoplatelets



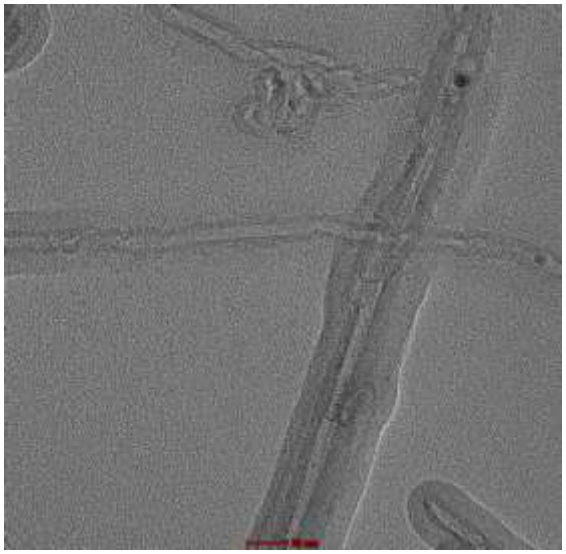
(c) Graphene nanoplatelets



(d) MWCNTs type 5



(e) MWCNTs type 5



(f) MWCNTs type 5

Fig 2.2: TEM image of graphene nanoplatelets and MWCNTs type 5 fillers.

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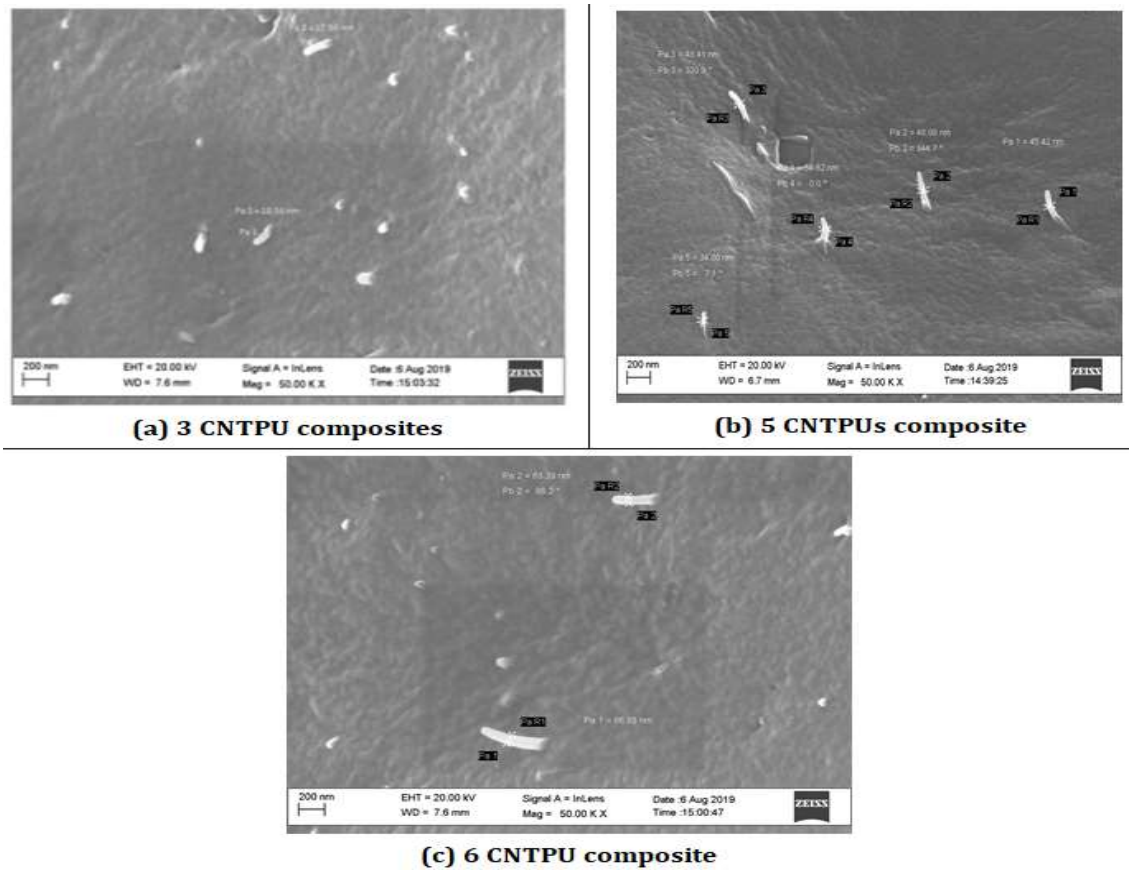

Fig 2.3: FE-SEM cryogenic fracture surfaces morphology of composite containing 1.5 phr MWCNTs into SMTPU matrix of (a) 3 CNTPU, (b) 5 CNTPU, (c) 6 CNTPU.

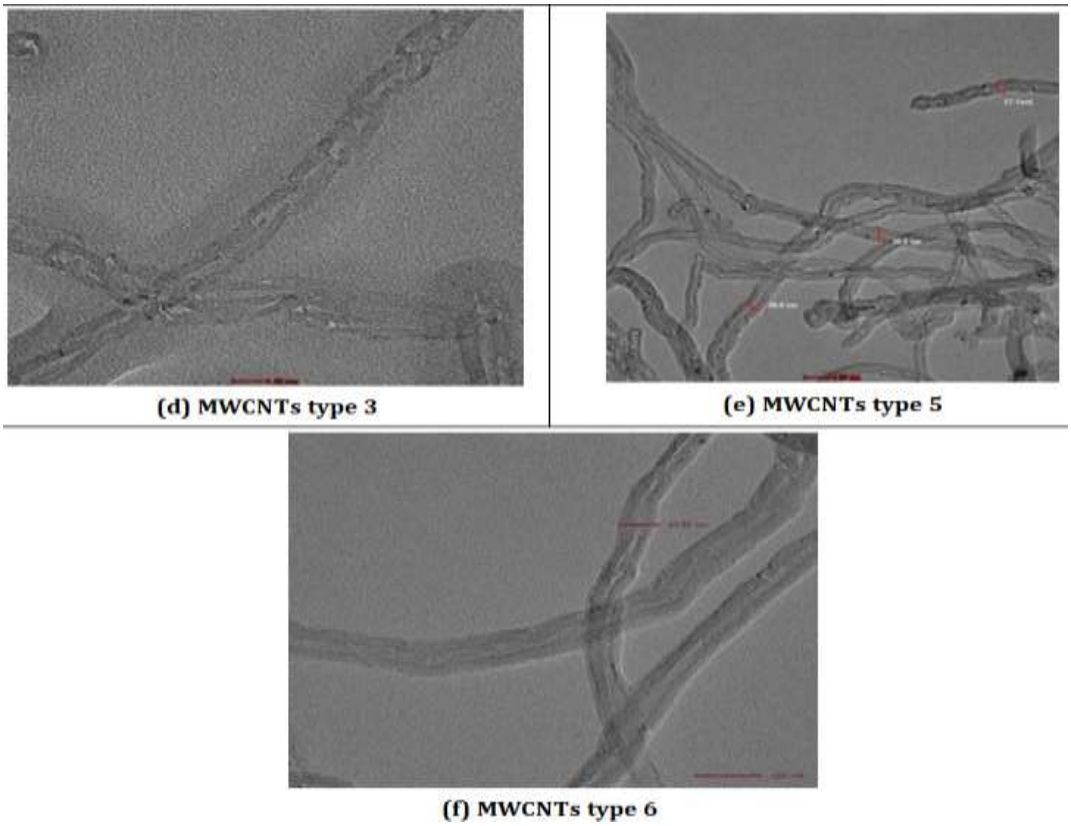

Fig 2.4: TEM image showing the diametres of (d) MWCNTs type 3, (e) MWCNTs type 5 and (f) MWCNTs type 6.

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2.3 X-Rays diffraction (XRD): X-Rays Diffraction (XRD) analysis were done with use of XRD 6000 X-rays diffractometer (Shimadzu, Tokyo, Japan) 30 mA, 40 kV, λCuKα = 0.15 nm with high diffraction angle 2θ varied from 0°–100° at the scan rate of 2°/min.

X-ray technique was used to examine peaks of fillers as well as base matrix and the interfacial interaction between SMTPU and fillers materials. Fig. 5.6 shows the XRD of (a) SMTPU, MWCNT type 5 and its composites, (b) of SMTPU, GNPs and its composites, and (c) of SMTPU and composites containing 1.5 phr MWCNTs type 5 , (type 3, and type 6). The peak of MWCNT occurs at d_{002} and d_{100} the angles (2 theta) of 25.8° and 42.6° respectively. Peak d_{002} was higher because crystalline natures of MWCNT powders and d_{100} is smaller because the amorphous phases of MWCNT. Pure SMTPU peaks have observe as d_{100} at 2θ=20.5° and small peak also observe at 2θ= 28.0°. d_{100} peaks are broad due to the amorphous natures of polyurethanes. Small peaks occur due to partial crystalline nature of polyurethane. The peaks of CNTPUs composite samples show at d_{100}, d_{002} and d_{004} which indicate proper mixing of MWCNT into SMTPUs matrixs. The peaks about 25.8° goes higher with the addition of higher percentage of MWCNT into SMTPU matrix (Fig. 5.6 (a)). Single peak d_{002} of GNPs occur at 2θ=25.8°, because graphene nanoplatelets are highly crystalline materials. Fig. 5.6 (b) shows the peaks of GNPs and their polyurethane composites containing 0 to 1.5 phr GNPs. The peaks of composites observed at d_{100} and d_{002} with the angle of 2θ=20.5° and 2θ=25.8° respectively. With the addition of higher and higher percentage of GNPs into SMTPU matrix the peak of d_{002} at 2θ=25.8° going to increase. Similar observations were also concluded from Fig. 5.6 (c). Which shows the XRD of pure and composites sample of 1.5 phr MWCNTs (type 5, type 3, and type 6) into SMTPU matrix. Proper mixing of fillers into base matrix has been verified from the XRD analysis.

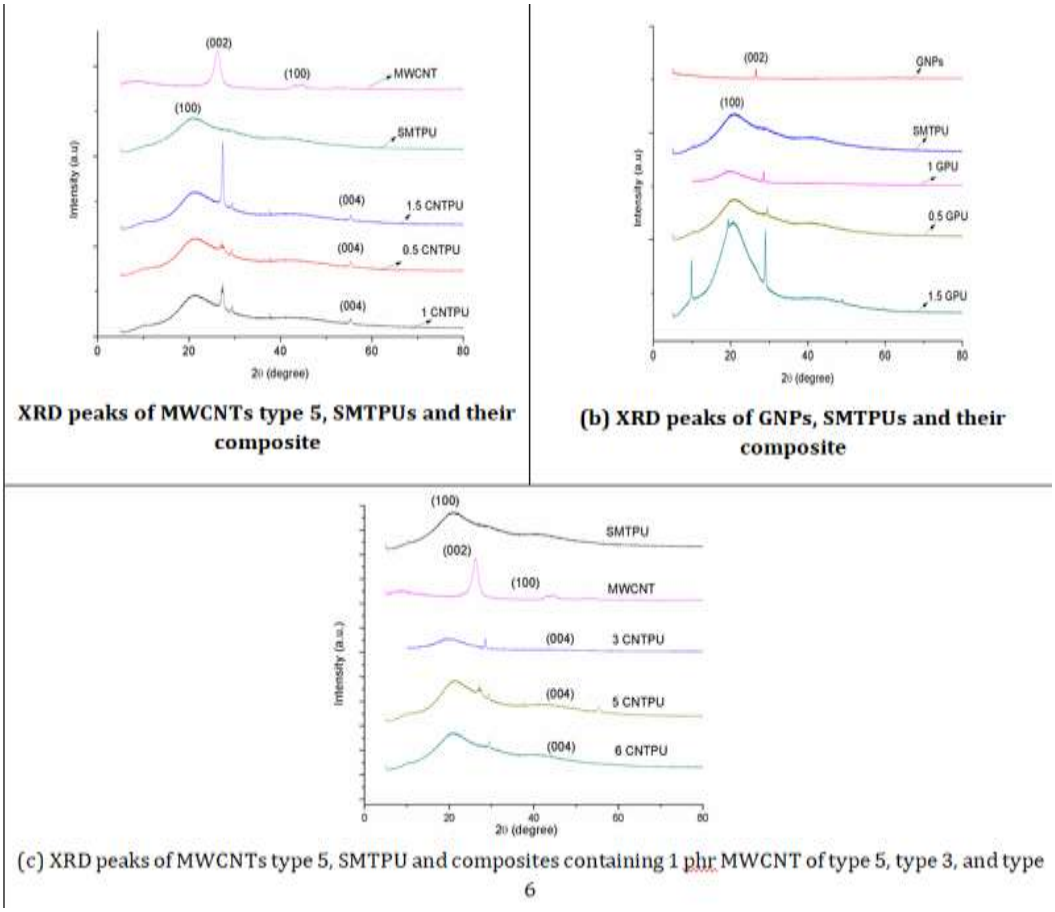

Fig 2.5: X-rays diffraction peak of MWCNTs, GNPs, pure and composites sample.

2.4 Atomics Force Microscope (AFM): AFM is high resolutions microscope, through which microstructure of any material can be studied. AFM is an improvement to STM (Scanning Tunnelling Microscope), as in STM only conducting surfaces were analyzed whereas in AFM any and every material is fit to be used for microstructural investigation . Here AFM is used for clear characterization of surface morphology like surface roughness,

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waviness, and dispersion of GNPs in PU matrix. Fig.5.7 shows the AFM surface characterization of 2D and 3D images of GNPs, MWCNTs type 5 and pure SMTPU samples. Fig. 5.7 (a, b) shows shape and size of GNPs and MWCNTs type 5 respectively. Firstly the powder of GNPs and MWCNTs were coated in silicon befalls through spin coating technique for proper characterization. The surface area of GNPs was observed to be about 50-80 m^2/g.

III. CONCLUSION

In present research work, involve fabrication of multiwalled carbon nanotubes (MWCNTs) (MWCNTs type 3, MWCNTs type 5 and MWCNTs type 6), reinforced into shape memory thermoplastic polyurethane (SMTPU) by use of melt mixings route by the use of micro-compounder followed by mini injection moulding. The composites of multiwalled carbon nanotubes (MWCNTs) were prepaired through ex-situ polymerization, into SMTPU pure matrix, containing 0 to 1.5 phr (par per hundred) fillers. Successfully dispersion was achieved for various percentage of nano fillers into SMTPU pure matrix which is characterized using Fields Emissions Scanning Electrons Microscope (FE-SEM), High Resolution Transmission Electron Microscope (HR-TEM), Atomic Force Microscope (AFM), and X-Rays Diffractions (XRD) etc. the shape and size of nano fillers (MWCNTs and graphene nanoplatelets) was characterized using high quality TEM and FE-SEM, and AFM techniques.

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