Microwave–Induced Thermo-Responsive Shape Memory Polyurethane/MWCNTs Composites and Improved their Shape Memory and Mechanical Properties

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ABSTRACT

Microwave (MV)-induced thermo-responsive shape memory thermoplastic polyurethane (SMTPU)/ MWCNT composites were prepared in micro-compounder. Composites containing different amount of multiwall Carbon nanotube (MWCNT) varying from 0 to 1.5 phr in SMTPU matrix were prepared. Maximum stretching strength, recovery force and tensile strength for 1.5 CNTPU (1.5 phr MWCNT in SMTPU matrix) was increased by 120%, 100% and 24% respectively as compared to SMTPU. MV-induced shape memory is a novel approach for fast, clean and remote heating during operation. MWCNT is strong absorber of microwave irradiation so that SMTPU/ MWCNTs nanocomposites successfully triggered by microwave.

KEYWORDS : *Shape memory polymer; Microwave, MWCNT, Thermal Imager.*

1. INTRODUCTION

Shape memory polymers are smart, intellectually, functionally graded, self-healing, lightweight, low cost, ease of fabrication as compared to existing shape memory alloys and shape memory ceramics [1-5]. As exploring new research in science and technology day by day, the shape memory polymer nano-composite components take place over the existing components[2,6,7]. Shape memory polymers (SMPs) usually used for various applications such as sensors and actuators, remote sensing and wireless, bio-medical devices, robotics and other sophisticated applications. Shape memory materials are the class of self actuating materials which are switching the one temporary shape to other permanent shape with the help of number of external stimuli such as,

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temperature[3], magnetic field[8], electric current, ph[6-7], water[9], solutions, electromagnetic rays[10], microwave etc[11]. In the shape memory polymer (SMP) the phase is changing below and above the glass transition temperature (Tg). Below the glass transition temperature the shape memory polymer components behave like a glass (hard phase) which is called the glassy state and above the transition temperature its behave like a rubber called as a rubbery state. In shape memory polymer generally two phases come into the picture one is a soft phase and another is a hard phase[6,12,13,14]. Soft phase is only responsible for switching the temporary to permanent and permanent to temporary shape in shape memory polymer below and above its glass transition temperature. Shape memory effect shows at glass transition temperature[15-17]. With increasing the utility of self-actuating devices, the shape memory polymer components may fulfil those requirements. Shape memory polymer components are widely used as compared to shape memory alloys components because of ease of fabrication, high strain rate (upto 800%), low processing temperature, light in weight, excellent corrosion and electrical resistance, bio-compatible etc[13,18]. on other hand certain disadvantages are also in shape memory polymer as compare to shape memory alloys such as low recovery strength, low stretch strength, low operating temperature[2], easily affected by moisture[9], etc. for improving the shape memory and mechanical properties of shape memory polymer various fillers such as fibre, SiC, graphene, GNPs, CNTs were used[19-20]. Amongst these, Carbon-based nanofiller such as graphene, GNPs, and CNTs are excellently pronounced reinforced for shape memory polymers due to their excellent mechanical, thermal, and electrical properties. So that for improving shape memory and mechanical properties of polymer-based shape memory components most vital available CNTs and graphene nanofillers are used[17, 21, 22]. Various researchers also investigated that the multiwall Carbon nanotubes (MWCNTs) were reinforced in shape memory thermosetting polyurethane (SMPU) for improving the shape memory and mechanical properties. In various literature surveys, we found that the shape memory components are generally triggered by various stimuli such as temperature, water, magnetic current, ph, and solution [2]. When we think about the non-conventional, non-contact heating during the operating devices only stimuli such as electromagnetic waves and microwaves (MV) are useful for various long distance (aerospace), wireless, remote sensing applications. Various researchers have already started work on electromagnetic and light-induced shape memory polymer composites[23-27]. Graphene, SiC, MWCNTs are strong microwaves and electromagnetic waves absorbent materials. Many researchers investigated that the photo-responsive triple way shape memory PU/azobenzene/graphene oxide (GO) composites film were prepared through a solvent casting route. Where GO and azobenzene act as a heat source in the PU matrix [28-30]. Photo-responsive shape memory polymer nanocomposites exhibit the low cost and volumetrically fast actuating[29]. UV and NRI light responsive shape memory polymer composites reinforce with CNT were investigated by researchers for fingerprint pattern write and erase applications. Our previous research also reported that MV-induced polyurethane/GNPs nano-composites were successfully trigged and properties were also improved with the addition of GNPs in the PU matrix[27,31,32]. Our previous research for improving shape memory and mechanical properties the graphene nanoplatelets (GNPs) were used in polyurethane matrix[33]. Microwave induced shape memory polymer composites components are volumetrically heated which may help for uniform and fast actuating applications.

In this paper, non-traditional, non-contact, fast, volumetric heating microwave induced shape memory polymer to reinforce with MWCNTs were used for improving shape memory and mechanical properties. An experimental study was carried out for different concentration of multiwall Carbon nanotubes (MWCNTs) were randomly distributed in shape memory thermoplastic polyurethane (SMTPU) matrix with the help of micro-compounder. Stretching strength, recovery force, tensile stress-strain, shape fixity, thermal diffusivity and shape recovery tests were studied and reported in this research. With the addition of MWCNTs in SMTPU matrix, the shape memory and mechanical properties were improved. Microwave heating has a several advantages over the conventional heating such as, volumetric heating, fast heating, the clean green source of heating, non-contact heating, low cost heating, ease of control etc. so that MV-induced SMTPU/MWCNTs composite has great applications for wireless, long distance, sensors and actuators [32,33-34].

2. EXPERIMENTAL DETAILS

2.1 Materials

Shape memory thermoplastic polyurethane (SMTPU) (ether type) granules MM6520 purchase from, SMP Technology Inch. Japan (Mitsubishi). Multiwall Carbon nanotubes (MWCNTs) type 5 having outer diameter 30-50nm length 30-50μm is purchase from Sisco Research Laboratories Pvt. Ltd.

2.2 Sample preparation

MWCNTs powder and SMTPU granules firstly dried in a vacuum oven at 100°C for 5 hours to remove the carbonaceous impurities. A Thermo Haake MiniLab 3 (model PolyLab OS) micro-compounder having twin conical screw was used for melting and simultaneous mixing the MWCNTs and SMTPU. The rotor speed and mixing temperature of micro-compounder was 60 rpm and 210°C respectively for ten minutes. The SMTPU/MWCNTs specimen having different phr (0 to 1.5) namely, SMTPU, 0.5 CNTPU, 1 CNTPU and 1.5 CNTPU were prepared in micro-compounder through the melt mixing route. In micro-compounder 6g SMTPU and MWCNTs compositions were mixed. After that melt mixture was taken in the cylinder which is already heated at 210°C and processed for Injection Moulding, An injection moulding machine Thermo Scientific Germany (HAAKE Mini-jet Pro Piston) was used for samples preparation. The melt mixture from micro-compounder is directly injection moulded in desired shape and size (ISI slandered SS die) at moulding temperature 210°C, injection pressure 500bar, injection time 6 seconds and mould temperature 85°C. Then after 5-8 minutes, the samples removed from the die for testing and characterizations.

2.3 Tensile testing

The tensile tests were performed on a (Tinius Olsen25 kt) tensile testing machine with wedge clamps. The tensile tests were conducted at room temperature and speed of cross-head was 10 mm/minute. The tensile samples size was standard ISI tensile die, prepared indirectly from injection moulding die.

2.4 Shape memory test

The shape memory stretch and recovery tests were performed using the Tinius Oslen25kt tensile testing machine with wedge clamps. In the shape memory stretch test the sample size, 50x10x1 mm^3 was clamped in wedge shape spring-loaded grip and heated at 65°C. At 65°C, the force is applied at the rate of 2 mm/minutes upto 100% strain. Thereafter the furnace chamber is open to cool the sample below 32°C without releasing the load. Once the sample cools below 32°C the sample removes from the grip and measure the final gauge length for shape fixity. And after measuring the shape fixity the stretched sample again clamp in the grip for measuring stretch recovery force. For a stretch,recovery tests the stretched sample clamp in grip and heating the sample to 65°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. Detailed stretch and recovery tests were discussed in the results and discussion section.

2.5 Field Emission Scanning Electron Microscopy (FE-SEM)

Cryogenics fractured surface was analysis by using (SEM of M/s NanoSEM 430). The liquid nitrogen was used for cryogenic fracture the samples and fractured samples were gold coated at 100 A^0 for clear observations of surface morphology.

2.6 Dynamic Mechanical Analyzer (DMA)

In Dynamic Mechanical Analysis (DMA) thermo-mechanical properties of visco-elastic materials were studied by using DMA (DMs 6100 by Hitachi Instruments). The glass transition temperature (Tg), storage modulus (E'), loss modulus (E") and energy dissipation factor (Tan D) were studied. The injection moulded sample size 40X10X1 mm^3 were tested in three-point bending mode having frequency 1 Hz, heating rate 2°C/minutes in the temperature range from 30°C to 80°C.

2.7 Thermal Diffusivity

Thermal diffusivity was tested in liquid nitrogen chamber ranging from 25°C to 75°C having samples size 16.5mm diameter and 0.5 to 1.5 mm thickness by using Thermal Analyzer NETZSCH, Germany (LFA467 Hyper Flash).

2.8 Microwave-Induced Shape Recovery Test

MV-induced shape recovery test was conducted by using household microwave oven (IFB Model: 30SC3) having constant frequency 2.45 GHz, the distance between the permatron was 30 cm, supplied by Technical System Pvt. Ltd. The shape memory tests were conducted in fully microwave mode, 50% MV+ Convection mode and Convection mode. In the MV-induced shape recovery test, the samples were temporarily deformed from "S (spring)" shape to straight shape. Shape recovery behaviour was captured by using the thermal imager.

3. RESULTS AND DISCUSSION

The Result of the tensile stress test was shown in Fig. 1. From Fig. 1 we concluded that the tensile strength is directly proportional to the loading concentration of MWCNTs in SMTPU matrix. With the addition of multiwalled Carbon nanotubes in SMTPU matrix, the tensile strength is increasing, which helps the superior shape memory for various applications. The maximum tensile strength for 1.5 CNTPU is 68 MPa which is 24% higher than the SMTPU specimen. The yield strength for SMTPU, 0.5 CNTPU, 1 CNTPU and 1.5 CNTPU is 53 MPa, 56 MPa, 63 MPa and 68 MPa respectively.

We also concluded that the toughness and yield strength were increasing with the addition of more MWCNTs in SMTPU matrix. Toughness can be defined as the area below the curve in the stress-strain graph. Increased toughness means the strain energy stored also increased which strongly promote the fast shape recovery and high tensile strength. A similar observation was reported by[33] in SMP/GNPs composites.

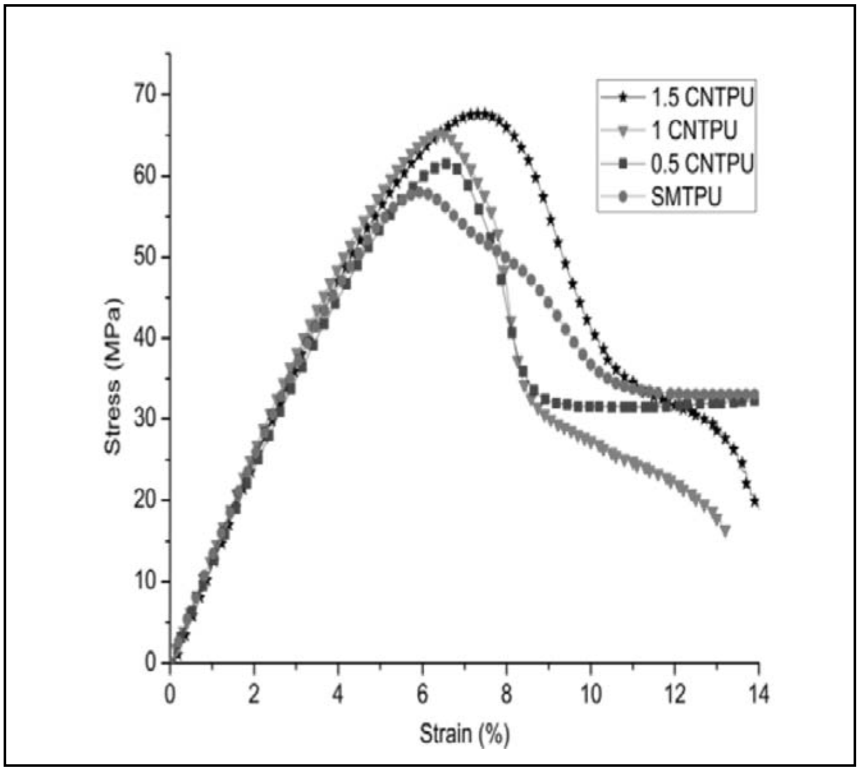

Fig. 1. The tensile stress-strain curve of SMTPU/MWCNTs composites

Fig. 2 shows the MV-induced shape recovery analysis of 1 CNTPU composite at 2.45 GHz constant frequency. The microwave induced shape recovery response of 1 CNTPU composite was too fast as compared to 50%MV+Convection and convection heating. Microwave heating of composites is very fast because the embedded MWCNTs in SMTPU matrix absorb the MV radiation and converted into the heat. MWCNTs within the matrix act as a heat node for specimen which is responsible for shape recovery in MV-induced shape memory test. Another hand, for convection heating the specimen heated as conventional heating. In a microwave, volumetric heating phenomena occurred which promote the fast heating of specimens. MV-induced shape recovery for 1 CNTPU specimen recovery it's approx 90% original shape within 40 seconds which is too fast as compared to convection heating. Whereas in convection heating for 1 CNTPU specimen take 100 seconds to recover its pre-deformed shape. A thermal imager was used to capture the shape recovery of 1 CNTPU specimen under the microwave heating which is shown in Fig. 3. For the shape recovery test, initially, the sample was temporary straight and sample put inside the microwave oven. After that, the microwaves start and sample starts to recover its pre-deformed “S spring” shape within 60 seconds. During microwave irradiation, the sample got heated because the embedded MWCNTs in SMTPU matrix act as heat node source under the MV condition. Similar observations we [33] also reported for SMP/GNPs composites specimen under MV-induced shape memory polymer.

Microwave induced shape recovery tests were conducted by using the household microwave having a constant frequency of 2.45 GHz. From Fig. 4 clearly shows that shape recovery speed of composites was drastically increased with the addition of MWCNTs in SMTPU matrix. The increased MV-induced shape recovery of composites because the embedded multiwalled Carbon nanotubes in SMTPU matrix absorbed MV irradiation and got volumetrically fast heated which is responsible for fast shape recovery. MV-induced shape recovery of 1.5 CNTPU sample is too fast as compared to SMTPU sample. For MV-induced shape recovery test the samples initially deform its temporary straight shape and after shape recovery, it’s come back to its original “S spring shape” shown in Fig. 3. The shape recovery of 1.5 CNTPU specimen recovery it’s 99% original shape within 55 seconds only, whereas SMTPU sample recovers shape only 50% to take 70

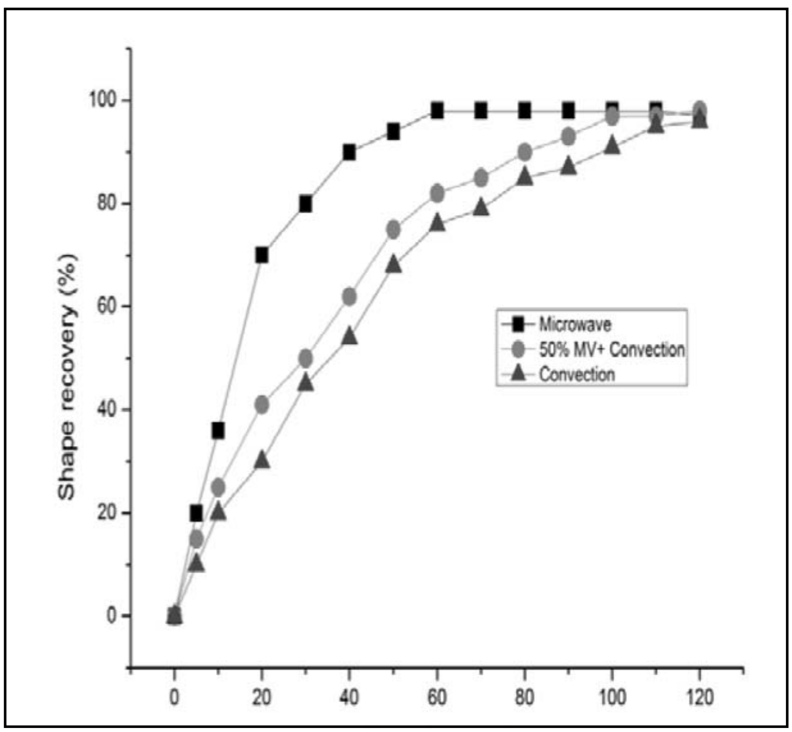

Fig. 2. MV-induced unconstrained shape recovery behaviours for 1 CNTPU at 2.45 GHz

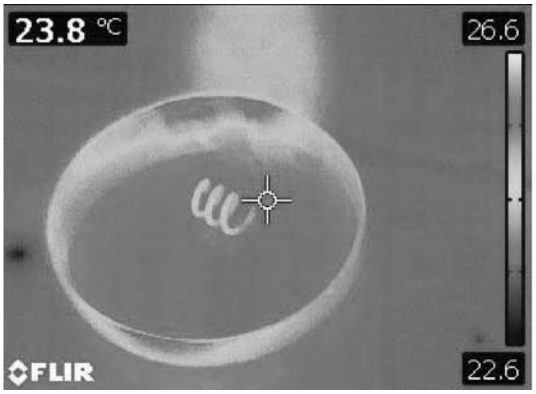

Fig. 3 (a) Permanent shape

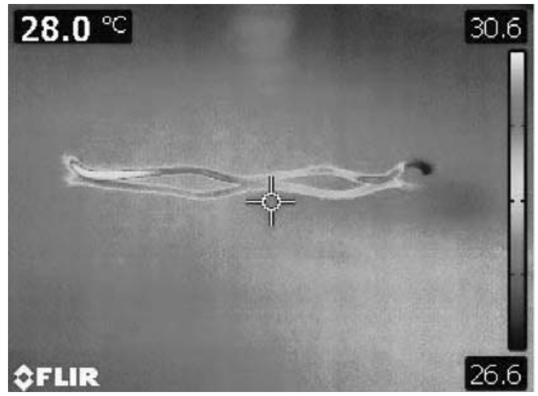

Fig. 3 (b) Temporary shape

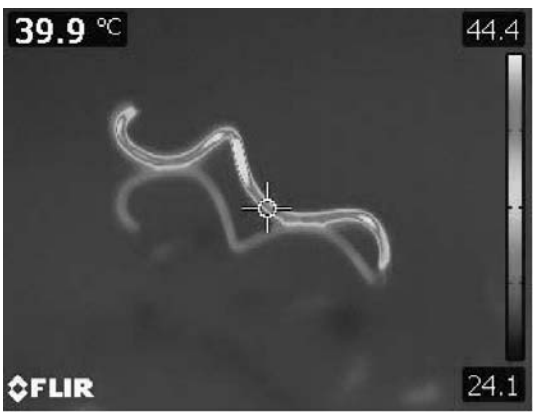

Fig. 3 (c) Shape after t=30 seconds

Fig. 4 (d) Shape after t=40 seconds

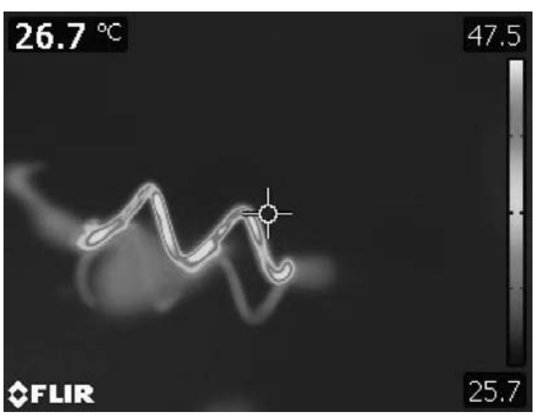

Fig. 5 (e) Shape after t=50 seconds

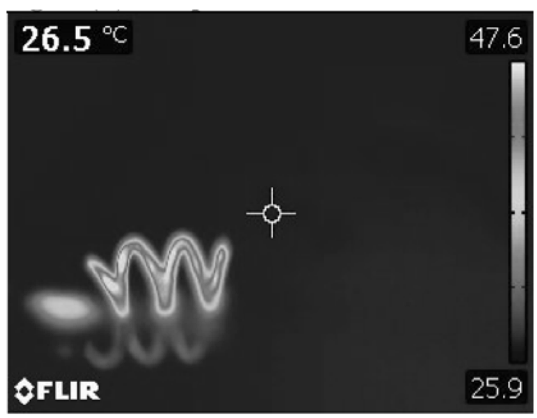

Fig. 5 (f) Shape after t=50 seconds

Fig. 3. MV-induced shape recovery test of 1 CNTPU specimen at 2.45 GHz

seconds shown in Fig. 4. Further, we concluded that the shape recovery also depends on the microwave frequency and loading concentration of reinforcement. Almost the same results were also reported by researchers [16,27,33] (SMP/GNPs composites and SMPU/CNT).

The shape memory stretch stress, recovery force and shape fixity tests were done under controlled hot chamber in the tensile testing machine. For stretch strength testing the tensile specimen was clamped between the spring-loaded grips and furnace start for heating the sample upto 65°C. Once the temperature of specimen reached 65°C the test was started and applied stress recorded to the 100% strain. After that, the furnace was stopped and waits for cooling the specimen below 32°C for measuring the shape fixity. The initial and final gauge length was a measure for shape fixity. Therefore, after measuring the shape fixity the stretched sample again loaded in the grip for a constrained recovery force test. For constrain recovery force test, sample heated at 65°C and applied force was simultaneously recorded at 65°C. After sometime within 60 minutes the recovery force was almost constant, thereafter the test was stopped.

From stretching stress test graph, it clearly shows that the stretching strength was improved with the addition of more multiwalled Carbon nanotubes in SMTPU matrix. Increase the strength of composites due to the MWCNTs

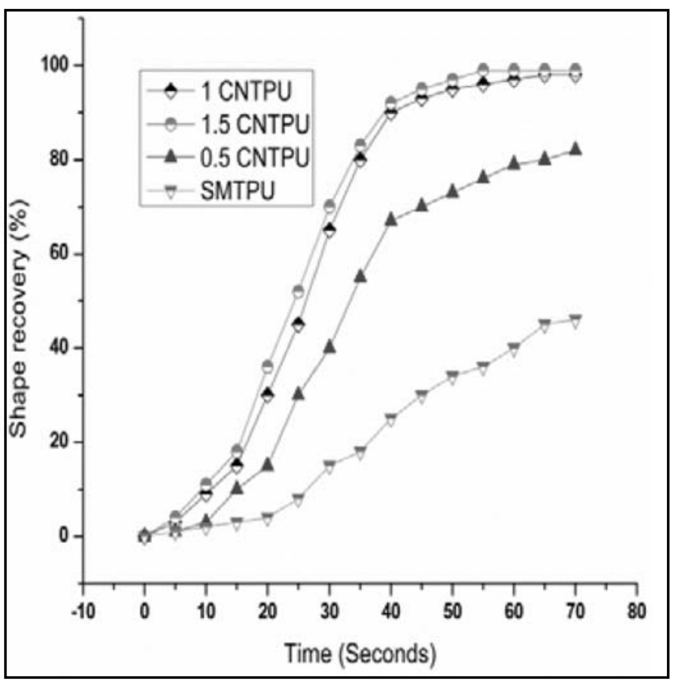

Fig. 4 MV-induced shape recovery test of SMTPU/MWCNTs composites (microwave oven at 2.45 GHz frequency).

filler having a high strength which helps the superior mechanical properties of SMTPU composites. Embedded nanotubes in composites stored the stretch energy in the form of strain energy during the stretching which is strongly useful for increasing the recovery force and shape fixity. The maximum stretch strength for 1.5 CNTPU composite is 2.2 MPa which is 120% higher than the SMTPU. The maximum stretching strength of SMTPU, 0.5 CNTPU, 1 CNTPU and 1.5 CNTPU is 1.1 MPa, 1.6 MPa, 1.8 MPa and 2.2 MPa respectively. Similar observations were also reported by various researchers [16,33].

Fig. 6 shows that the recovery force was enhanced with the help of MWCNTs reinforce in SMTPU matrix. As long as the percentage of MWCNTs in SMTPU matrix was increasing the recovery force was also increased. The maximum recovery force for 1.5 CNTPU was 8 N which is almost 100% more than the SMTPU.

Shape fixity (R_f) is an important parameter in shape memory tests, shape fixity define as the ability to fix a temporary shape during cooling below Tg and maintain that shape until the external stimuli not applied such as, temperature, light, magnetic and electrical fields, and PH etc. mathematically it is express as the ration of fixed strain (ϵ_f) to the maximum deformed strain (ϵ_d). $R_f = \epsilon_f \div \epsilon_d \times 100$

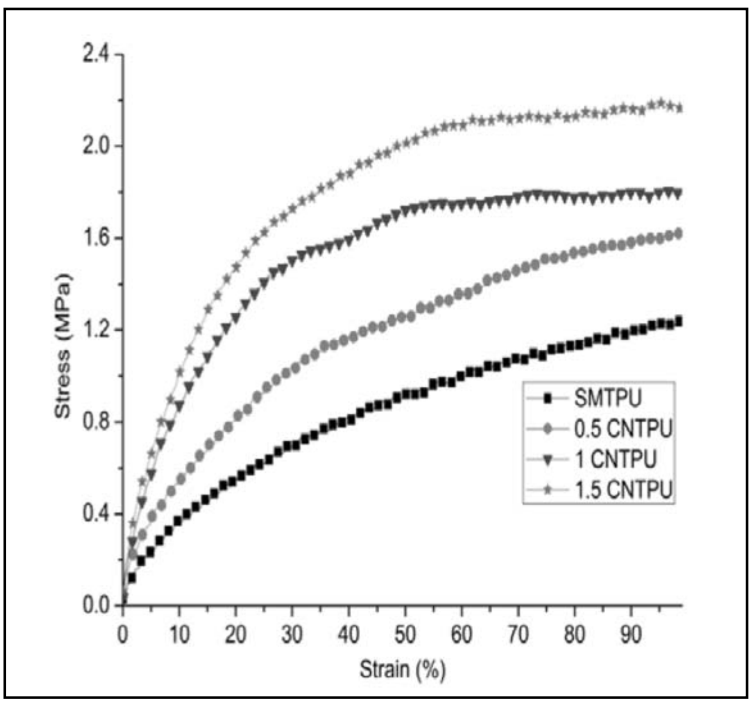

Fig. 5 Shape memory stretching stress (100% stretch) of SMTPU/MWCNTs composites

ϵ_f and ϵ_d were calculated as mentioned below;

$$\epsilon_f = (L_2\text{-}L_0)/L_0 \text{ and } \epsilon_d = (L_1\text{-}L_0)/L_0$$

Where, L_0 is gauge length, L_1 maximum length after deforming and L_2 is the length of the specimen when load removed[16].

Shape fixity of SMTPU/MWCNTs composites was shown in Fig. 7. The shape fixity was improved with the addition of MWCNTs in SMTPU matrix. Maximum shape fixity was observed for 1.5 CNTPU specimen. The shape fixity for SMTPU, 0.5 CNTPU, 1 CNTPU and 1.5 CNTPU were 94%, 96%, 98% and 99% respectively. Enhanced recovery force and shape fixity for composites because the strain energy stored by MWCNTs during the stretching. Improved shape fixity due to the addition of GNPs, SiC and MWCNTs in the PU matrix reported by researchers[16,27,33].

Fig. 8 shows that the cryogenic fractured surface morphology was studied after 100A^0 gold coating of SMTPU/MWCNTs composite specimen. SMTPU specimen have a more smooth and regular surface, whereas from Fig. 8(b) the smoothness and regularities were suppressed. Increased surface roughness was indicated that the uniform and random distribution of multiwall Carbon nanotubes in

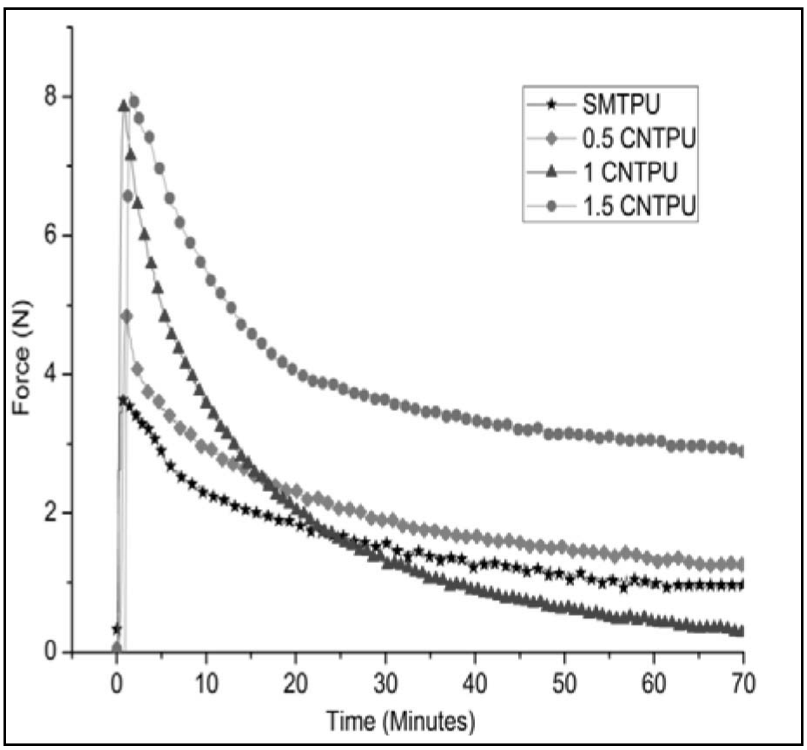

Fig. 6 Constrained shape recovery force of SMTPU/MWCNTs composites at 65°C

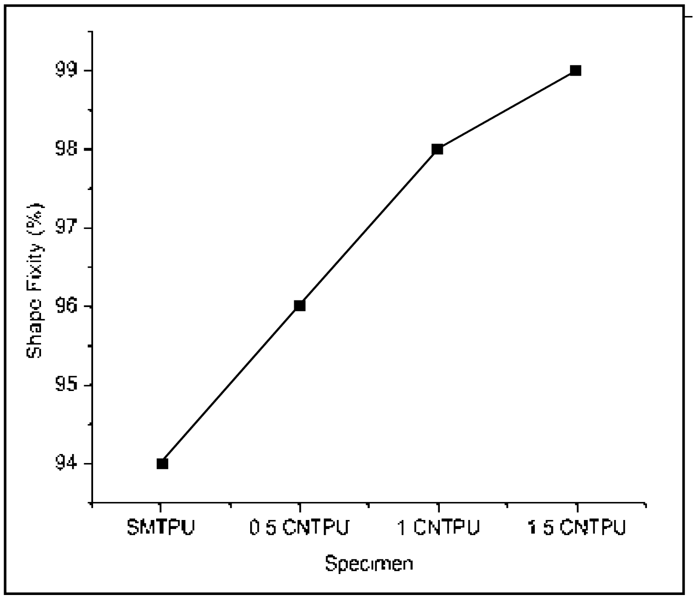

Fig. 7 Shape fixity of SMTPU/MWCNTs composites

shape memory polyurethane matrix. Random and homogeneous distribution of nanotubes may help for superior mechanical and shape memory properties of composites.

In Fig. 9 thermal diffusivity (α) of SMTPU/ MWCNTs composites were analyses (by using Thermal Conductivity Analyser) in temperature ranging from 30°C to 70°C. Thermal conductivity plays an important role in thermo-responsive shape memory. Thermal conductivity is directly proportional to the thermal diffusivity, which is shown by equation (1).

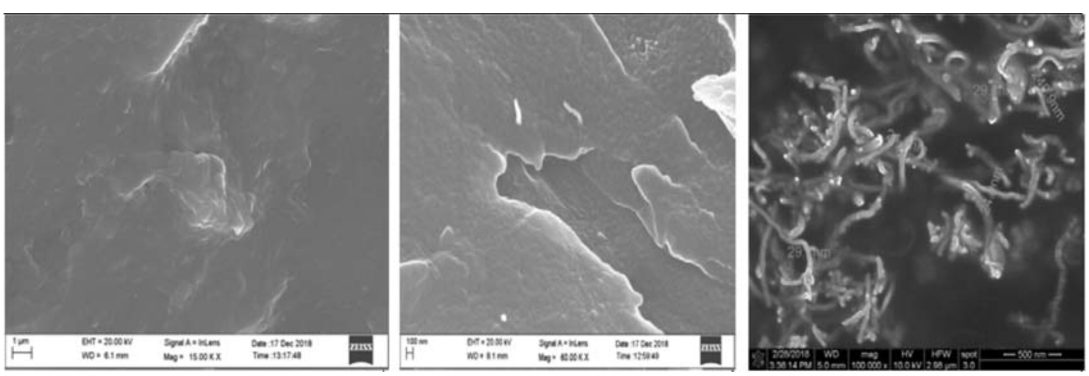

Fig. 8 Image showing cryogenic fractured surface after gold coating of (a) SMTPU (b) 1 CNTPU and (c) Multiwall Carbone nanotubes

$$\alpha = K / (\rho\ C_p) \qquad \text{Eq. (1)}$$

Where α = thermal diffusivity, K= thermal conductivity, ρ = density and C_p specific heat capacity

The addition of more MWCNTs in SMTPU matrix the thermal diffusivity was increased because of the embedded MWCNTs in SMTPU matrix acts as a heat conductor. On another hand, the thermal diffusivity decreased with continuous increasing temperature. The thermal diffusivity is decreasing with increasing temperature because the specific heat capacity (C_p) was increased. In Fig. 8 shows that the thermal diffusivity for 1.5 CNTPU specimen at 30°C is 15% higher than the SMTPU specimen. Improved thermal diffusivity of composites remarkably influenced the MV-induced shape memory properties of SMTPU/MWCNTs specimens. As long as the thermal diffusivity is increasing the shape recovery improved under the MV-induced shape recovery test. The similar observation we also reported for SMP/GNPs composites in previous research paper[33].

Fig. 10 shows that the energy dissipation factor (Tan D) curves of SMTPU/MWCNTs composites. Energy dissipation factor is a ratio of storage modulus to loss modulus. From energy dissipation curve we can say, peaks of energy dissipation factor decreased and shifted towards the higher temperature with the addition of more MWCNTs concentrations. The almost similar observation

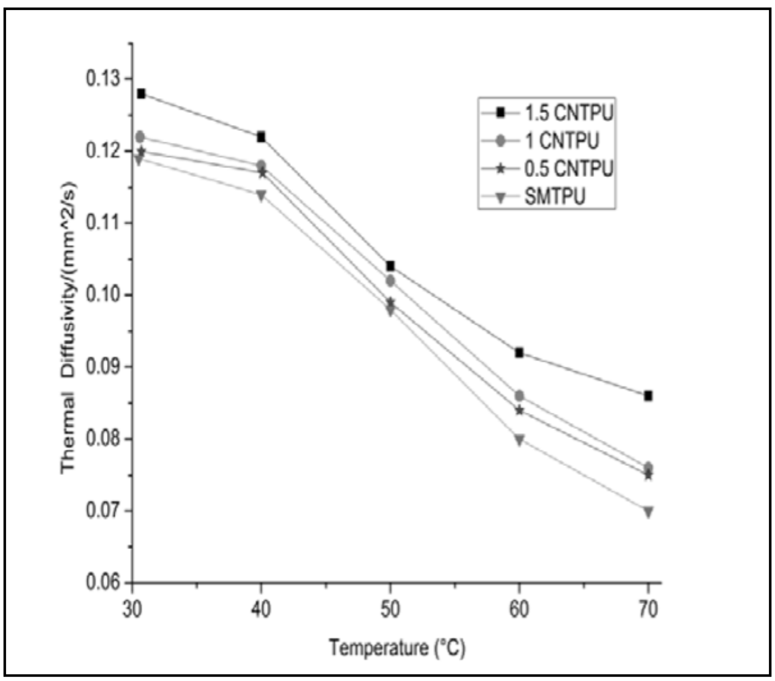

Fig. 9 Thermal diffusivity curves of SMTPU/MWCNTs composites specimens

was reported by[16,33]. With the addition of mare MWCNTs concentration in SMTPU matrix, the glass transition temperature was increased. Glass transition temperature was calculated by using the energy dissipation curves. Details of glass transition temperature was shown in Table 1.

The glass transition temperature is a key factor for shape memory polymer test. The glass transition temperature was strongly influenced by loading phr and dispersion of MWCNTs in SMTPU matrix. From Table 1 we concluded that the glass transition temperature is shifting toward higher temperature with the addition of a more MWCNTs concentration in the polymer matrix. Details glass transition temperature analysis was shown in Table 1 by using DMA curves.

TABLE 1. Glass transition temperature (Tg) calculation by using the DMA curves.

	SMTPU	0.5 CNTPU	1 CNTPU	1.5 CNTPU
Energy dissipation curve (Tg)	51	53	55	54

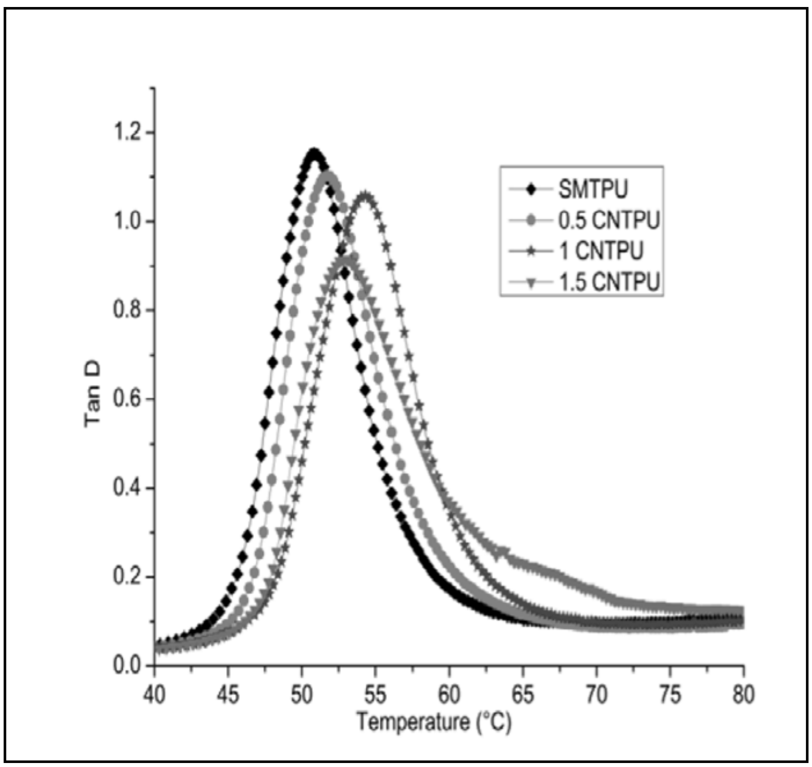

Fig. 10. Energy dissipation factor of SMTPU/MWCNTs composites

4. CONCLUSION

SMTPU/MWCNTs composites were successful trigged by microwave irradiation for fast, clean green, non-contact heating source, various wireless sensors and actuators applications. With the addition of MWCNTs in SMTPU matrix, the mechanical and shape memory property was improved significantly. The maximum stretch stress, recovery force, tensile strength and loss modulus for 1.5 CNTPU is increased by 120%, 100%, and 24%, and respectively as compared to SMTPU specimen. With the addition of MWCNT in SMTPU matrix, energy dissipation (Tan D) and loss modulus curve peaks shifted towards the higher temperature which we can say, the glass transition temperature was increased with the addition of MWCNT in the matrix. MV-induced shape recovery response was improved with the addition of MWCNTs because of the embedded MWCNT in SMTPU matrix act as a heating node source. The 1.5 CNTPU recover its 99% original shape within60 seconds, whenever SMTPU take 70 minutes to recover its only 50 % original shape.

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