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THERMAL PERFORMANCE OF SHAPE MEMORY THERMOSETTING POLYURETHANE COMPOSITE

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Abstract: This paper investigates the effects of Multi-walled-carbon-nano-tube on the thermal behavior of a polyurethane shape memory polymer. The effect of MWCNT reinforced polyurethane composite on shape memory behavior; glass transition temperature, tensile and recovery stress were measured. The shape memory behavior, tensile stress and recovery stress were measured by using conventional thermo-mechanical cycle. Loading of MWCNT reinforcement in the polyurethane composite increases the tensile stress, recovery stress, glass transition temperature and also improved the shape memory effect as compare to pure polyurethane.

I. INTRODUCTION

The past few decades has seen that the development of several new and novel materials for improved performance in engineering applications. Among them there is a class of materials which can sense and respond to their environment by changing their chemical or physical properties. These materials have attracted material scientists and engineers due to their interesting properties and potential applications. The materials having such unique property are known as smart materials or Stimulus Responsive Materials. Shape memory polymers have drawn a great attention from the research communities for its unique feature in the recovery of its original shape by a certain stimuli like heat, light, moisture etc [1-4]. The shape memory materials are stimuli-responsive materials having ability to undergo a large deformation and recover their original permanent shape upon the application of external stimulus. Many types of materials like alloys, polymers, ceramic and gels are known to exhibit the shape memory effect. Among them Shape Memory Alloys (SMA) and Shape Memory Polymer (SMP) are extensively studied at present. Temperature-responsive shape memory polymers or thermo-responsive shape memory polymers are polymers which exhibit a drastic change of their physical properties with temperature. Thermo-responsive polymers belong to the class of stimuli-responsive materials, in contrast to temperature-sensitive (thermo-sensitive) materials, which change their properties continuously with environmental conditions. Shape-memory polymers (SMPs) are polymeric smart materials that have ability to return temporary shape to their original permanent shape induced by an external stimulus, such as temperature, light, moisture etc [5-9]. SMPs can retain two or sometimes three shapes, and the transition between those is induced by temperature. In addition to temperature change, the shape change of SMPs can also be triggered by an electric or magnetic field. As well as polymers in general, SMPs also cover a wide property-range from stable to biodegradable, from soft to hard, and from elastic to rigid, depending on the structural units that constitute the SMP. SMPs include thermoplastic and thermoset (covalently cross-linked) polymeric materials.

II. SAMPLE PREPARATION

The shape memory thermosetting polyurethane consist of two part 1) Polyurethane resin (A) and 2) Polyurethane hardener (B). C is the reinforced solution of 0.5gm MWCNT and 19.5g of solution (B). The solution C was homogeneously mixed by using simultaneous ultra-sonication and magnetic stirrering at room temperature for 20hrs. MWCNT mixed hardener(C) as well as resin (A) and hardener (B) was kept under vacuum separately in a glass beaker for 2.5 hr. Mixing the solution A, B and C in appropriate amount within 30sec and again degassing the mixed solution for further 90 second and finally casting the mixture in mould. Samples were removed after 24hrs. The thicknesses of the sample were 2mm width 5mm and gauge length 17 mm. Samples containing different amount of MWCNTs were prepared and were designated as SMTPU, SMTPUCNT-1, SMTPUCNT-2, and SMTPUCNT-3for 0.0, 0.5, 0.1 and 0.15 phr MWCNT in SMTPU respectively.

III. EXPERIMENTAL SET-UP

THERMO-MECHANICAL ANALYSIS SET-UP

The shape memory test of the samples were examined in water bath because water can remove the heat from the hot grips of equipment and test samples, faster and more uniformly as compared to the air, due to the higher heat capacity of water. Heat capacity of water is 4.1813 Jg-1K-1 and of air is 1.012 Jg-1K-1. The set-up was developed by CSIR-AMPRI for the characterization of shape memory polymers. The components of the thermo-mechanical set-up are as follows;

i. WATER BATH: The entire thermo-mechanical cycle for characterizing Shape Memory Effects were performed in a water-bath.

ii. ELECTRIC HEATER: The water bath is equipped with an electric heater with temperature controller. The electrical heater also contained a water circulatory system to maintain a uniform temperature trough-out the water bath. The electric heater was capable of heating the water bath at a rate of 5°C per minute.

iii. TENSILE LOADING APPARATUS: The loading apparatus consist of a fixed and a moving grip. The sample was clamped in between the two grips. The stretching force was applied to the upper movable grip via a loading screw.

iv. LOAD CELL: The stress developed in the sample is measured by a load cell fixed above the moving clamp. A load cell is a transducer that is used to convert a force into an electrical signal.

v. DATA ACQUISITION SYSTEM: The readings

from the load cell were obtained from the data acquisition system in the form of voltage-time curves.

3.3.2 ULTRASONIC PROCESSOR

Ultrasonic processor (sonicator) is used for homogeneous mixing of MWCNT in the SMTPU specimen. Ultrasonic electronic generator converts AC line power to 20000 Hz signal that powers a piezoelectric convertor/transducer. The transducer in turn converts this electrical signal to a mechanical vibration because of the attributes of the internal piezoelectric crystals. This mechanical vibration is intensified and passed down the length of the probe or horn where the tip expands and contracts longitudinally. The distance travelled by the tip depends on the amplitude chosen by the user via the amplitude control knob. In liquid, the fast vibration of the tip results in the formation and collapse of microscopic bubbles. This phenomenon is dubbed as cavitation. The breakdown of countless number of cavitation bubbles, releases significant amount of energy in the cavitation field. Following this, surfaces and objects inside the cavitation field are processed. Cavitation is caused for the sudden formation and collapse of high-pressure bubbles within the fluid. Extremely high pressure, temperature variations and shock waves are involved in the process. The diameter of the probe tip controls the amount of sample that can be suitably processed. Diameters of smaller tips i.e. micro-tip probes provide high intensity sonication; however the energy is directed inside a small, concentrated area. Although larger volumes are processed by larger tip diameters, they deliver low intensity sonication.

3.3.3 Differential scanning calorimetry (DSC)

The sample is placed in a suitable pan and sits upon a constantan disc on a platform in the DSC cell with a chromel wafer immediately underneath. A chromel-alumel thermocouple under the constantan disc measures the sample temperature. An empty reference pan sits on a symmetric platform with its own underlying chromel wafer and chromel-alumel thermocouple. Heat flow is measured by comparing the difference in temperature across the sample and the reference chromel wafers. Thermal characterization of samples of SMPU and MWCNT reinforced SMPU was conducted in DSC 1 STARe System of M/s Mettler Toledo. The heating and cooling rates were fixed at 3oCmin-1. The sample swere tested at a temperature range from 10oC to 70oC in a nitrogen atmosphere. The DSC curves of as prepared test samples were obtained for heating cycle.DSC is used to measure melting temperature, heat of fusion, latent heat of melting, reaction energy and temperature, glass transition temperature, crystalline phase transition temperature and energy, precipitation energy and temperature, oxidation induction times, and heat capacity.DSC measures the amount of energy absorbed or released by a specimen when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes.

IV. RESULTS

TENSILE AND RECOVERY STRESS

The shape memory test of the samples were examined in water bath because water can remove the heat from the hot grips of equipment and test samples, faster and more uniformly as compared to the air, due to the higher heat capacity of water. Heat capacity of water is 4.1813 Jg-1K-1 and of air is 1.012 Jg-1K-1. The tensile and recovery stress-strain curves of shape memory thermosetting polyurethane with various phr of MWCNT have been plotted by using conventional thermo mechanical cycle. Figures 1-4 show the tensile stress-strain and recovery stress-strain plots for SMTPU, SMTPUCNT-1, SMTPUCNT-2 and SMTPUCNT-3.

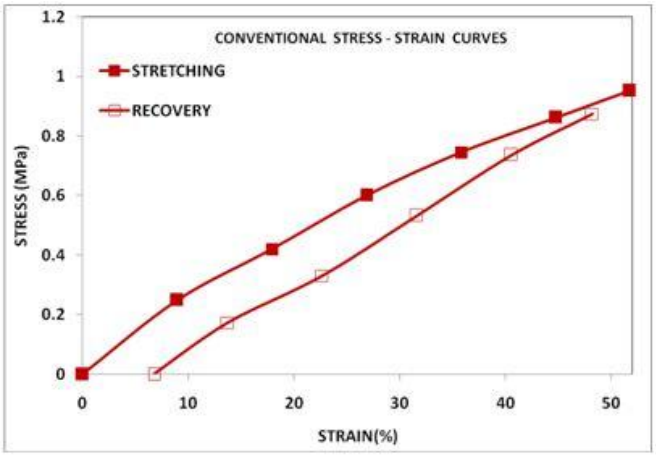

Figure.1: Stretch and recovery curves of SMTPU specimen at 65^{o}C.

The maximum strain was taken as 51% at 65^{o}C temperature in hot water environment.

i. Measure the gauge length of the sample. The thermo-mechanical cycle of thermo responsive shape consist following steps:

ii. Clamp the sample.

iii. Heating the sample above glass transition temperature.

iv. Deformed the sample by applying an external force.

v. Cools the sample bellow glass transition temperature.

vi. Remove the clamp to attain the temporary pre- deformed shape.

vii. Clap the deformed sample and heating above glass transition temperature (Tg) then measure recovery of the SMP towards its original shape.

The tensile stresses developed for the deformed specimen (51%) was 0.95MPa and the maximum recovery stress after fixing and reheating the specimen at this point was found to be 0.87MPa.

Similarly, SMTPUCNT-1, SMTPUCNT-2 and SMTPUCNT-3, were studied and stress-strain and recovery stress-strain plots were generated as shown in Figures2, 3 and 4 respectively. At strain 51% the tensile stresses developed in SMTPUCNT-1, SMTPUCNT-2 and SMTPUCNT-3, obtained 1.17, 1.74 and 2.18 MPa respectively. The recovery stresses at this point observed as 1.09, 1.65 and 2.00 MPa

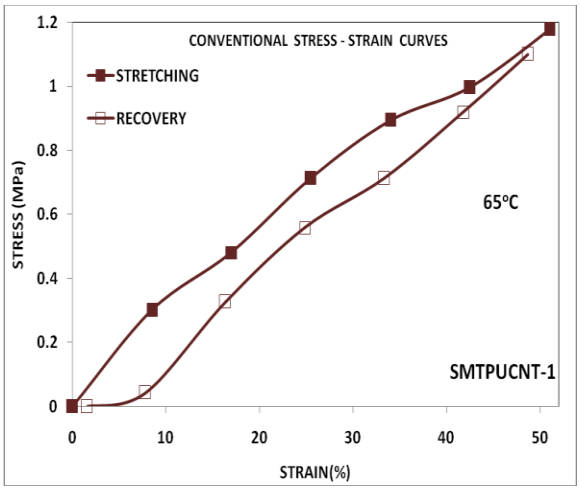

Figure.2: Stretch and recovery curves of SMTPUCNT-1 specimen at 65°C.

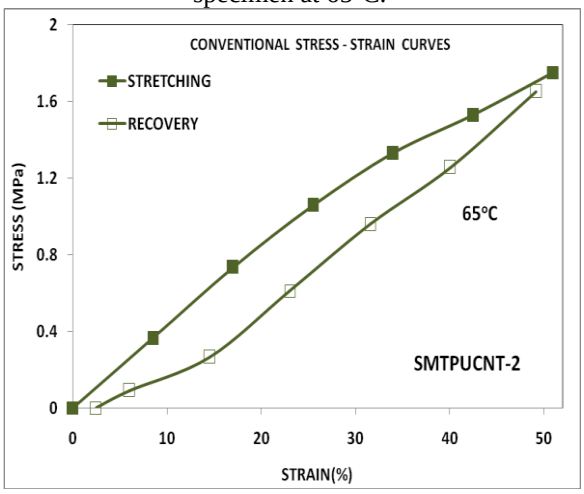

Figure.3: Stretch and recovery curves of SMTPUCNT-2 specimen at 65°C.

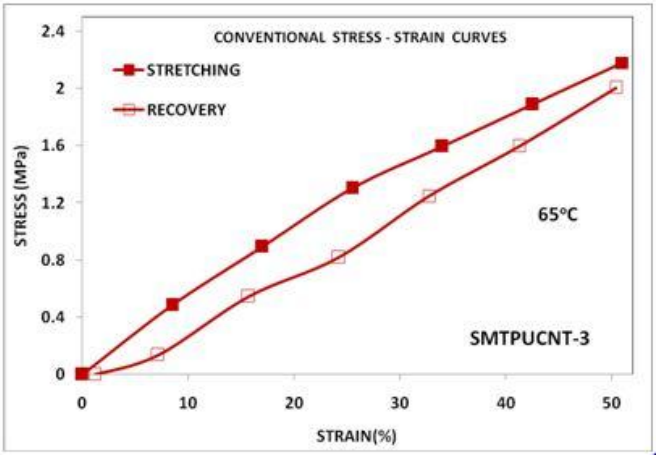

Figure.4: Stretch and recovery curves of SMTPUCNT-3 specimen at 65°C.

DSC is used to measure melting temperature, heat of fusion, latent heat of melting, reaction energy and temperature, glass transition temperature, crystalline phase transition temperature and energy, precipitation energy and temperature, oxidation induction times, and heat capacity. DSC measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic and exothermic processes.Figure.5 shows the DSC curve obtained by heating the samples from 10°C to 80°C at constant heating rate of 3oCmin-1 in the presence of nitrogen atmosphere.

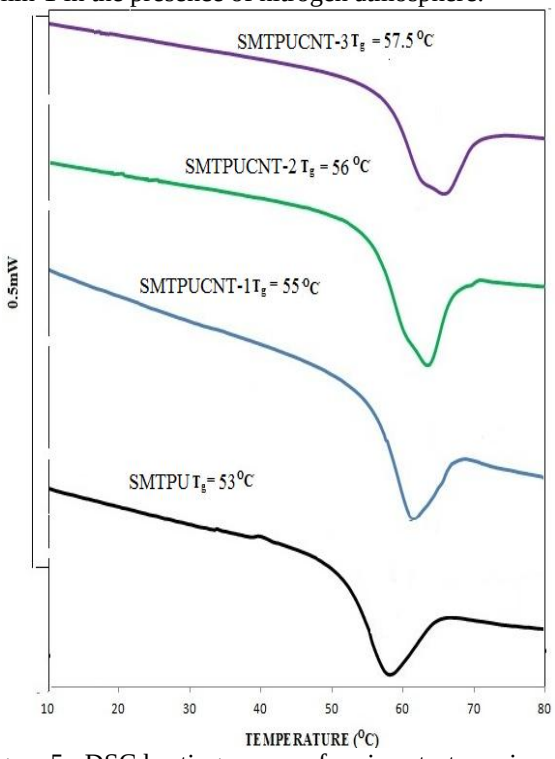

Figure.5: DSC heating curves of various test specimens.

Glass transition temperature (Tg) was taken as a median point in the glass transition range of DSC heating curve. The glass transition temperatures (Tg) of SMTPU composite were obtained. Glass transition temperature provides information about interactions of MWCNT with thermosetting polyurethane. Increasing the amount of MWCNT reinforcement in SMTPU samples improves the glass transition temperature of the composite. The improvement in Tg with reinforcement of MWCNT in SMTPU helped in improving properties of the composite.

FESEM analysis was carried out to understand dispersion state of nano-particle in the shape memory composite. Figure.6: shows the FESEM micrographs of pure MWCNT used in this study, wherein the majority of MWCNT were having diameter in the range of 30-50 nm. FESEM analysis was carried out to understand surface morphology and dispersion state of MWCNT in the SMTPU composite.

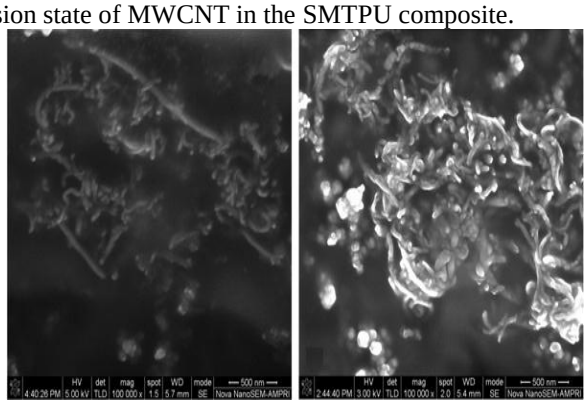

Figure.6: Micrograph of pure MWCNT.

V. CONCLUSION

In the present study shows that the MWCNTs are effective in improving the thermal, mechanical and shape memory properties of polymer composite. The properties of the MWCNT reinforced composite products are significantly affected by many factors such as processing techniques, filler distribution, sonication time, filler size, aspect ratio and matrix nature. In the experimental part of this study shape memory thermosetting polyurethane reinforced with multi-walled carbon nano-tubes were developed and characterized thermal and recovery stress analysis. The samples were studied for mechanical, thermal and morphological properties and for shape memory effects. The following can be concluded from the results obtained;

(i) Recovery time improved in MWCNT reinforced SMTPU composite as compared to pure SMTPU specimen.

(ii) The DSC studies showed that there was an increase in the glass transition temperature with the addition of MWCNTs in the polymer matrix.

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