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# Thermo-Mechanical Characterization of Unreinforced and Carbon-Reinforced Shape Memory Polymer

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## ABSTRACT

*Shape Memory Polymer (SMP) is a class of smart materials with unique capability of remembering its shape. It can be restored from a temporary fixed shape to permanent shape upon exposure to an external stimulus like heat, light, magnetic field, moisture etc. As opposed to its widespread use in many engineering applications, low stiffness and strength has restricted its excessive usage in structural engineering field. This research focuses on manufacturing and characterizing SMP and its carbon fiber reinforced composite (SMPC). The material is thermo-mechanically characterized for its transition temperature (Tg) using Dynamic Mechanical Analysis (DMA). Fold-deploy tests are conducted to determine shape fixity and shape recovery ratios. Additionally, the effect of temperature increase in recovery ratio is also investigated. Mechanical characterization is done via static uniaxial tensile tests. A substantial increase (100%) is observed in stiffness of SMPC compared to SMP. The samples are analysed for the effect of increasing shape memory cycles, which show that the major decline in stiffness essentially occurs after the first cycle. Similarly, on studying the effect of degree of deformation, the major decline in stiffness or increase in strength is found to occur after an angle of deformation of 45˚.*

**Keywords:** Composite, Shape memory, Stiffness, Strength, Polymer

## INTRODUCTION

Nature is full of instances with materials changing shapes in response to the fluctuations in their immediate environment [1]. Likewise, special functional polymers that have something unique to offer in terms of functionality have been the gauge of interest for quite some time. Shape Memory Polymers (SMP) are one such materials. They belong to a special class of materials viz. smart materials, particularly, to a subclass widely known as Shape Changing Polymers, which change the shape (or so does it appear macroscopically) in response to an external stimulus [2] like temperature [3-8], magnetic field [9-13], light [14-18], water [19-20], etc. This feature of shape memory polymers is known as Shape Memory effect (SME). Since this process involves change of shapes between two primary shapes (temporary and permanent) this effect is often termed as dual shape memory effect. The dual SME is illustrated in Fig. 1.

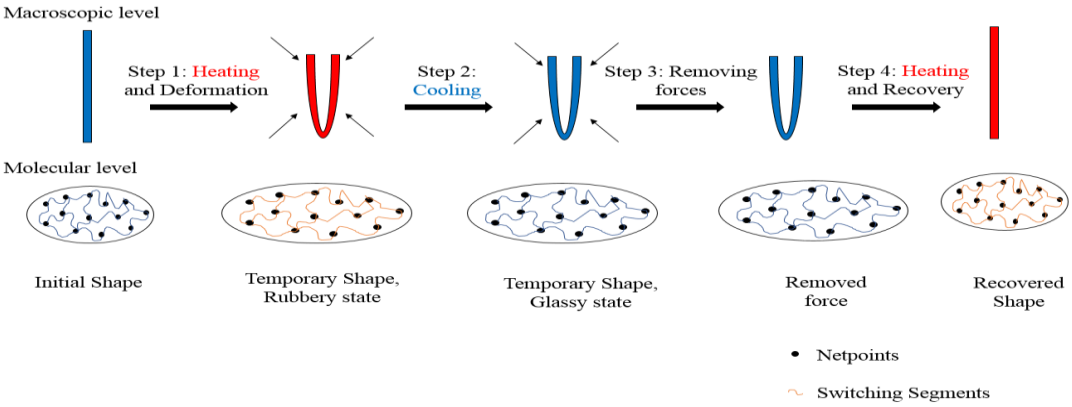



**Fig. 1 Schematic showing dual SME in shape memory polymers**

This exclusive SME property of these materials brings them on the forefront of materials to be used for numerous applications. Compared to other functional materials like electroactive polymer (EAP) or PZT, SMP is easier to process. Moreover, the ease of manufacturing, faster recovery rate, low cost and light weight properties of SMP over shape memory alloys have led to their rapid development and commercialization [3,21]. Until now, SMPs have had their share in wide ranging applications. It has been extensively used in medical field with the development of ophthalmic devices and its use in orthopaedic surgery [22]. Biomedical sector has also greatly benefitted from its usage [10,23,24]. Several industrial applications have also found their way with the development of shape adaptive grips, auto choke elements for engines etc. [25].

Various types of polymers have known to be exhibiting shape memory characteristics including epoxy based, cross-linked PE, cross-linked ethyl vinyl acetate polymer [26]. Out of the wide-ranging list, polyurethane (PU)-based SMPs (SMPU) have known to be advantageous on several fronts ranging from high shape recoverability, structure designability, wide range of glass transition temperature, and better biocompatibility [27]. SMPUs essentially exist in either rubbery sate or glassy state depending on the surrounding temperature. On a molecular level, it consists of rigid net points interlinked via flexible switching segments (or chains) (see Fig. 1). Net points determine the permanent shape and switching segments are responsible for fixing the temporary shape. On heating above its characteristic temperature (glass transition temperature, Tg; melting transition temperature, Tm; or less frequently used liquid crystal clearing temperature, Tcl) it becomes more flexible and easy to stretch/bend/curve. This flexibility is attributed majorly to the increased mobility of molecular chains. On the other side of the spectrum, below its transition temperature, it behaves as rigid plastic material and does not deform till sufficiently high loads are applied.

In the past, micro to nano scale fiber reinforcements have been added to improve thermo-mechanical and dynamic properties of SMP [28]. More so, hybrid fillers like chopped short carbon fibers and carbon black have been added to make insulating shape memory polymer conducting and evaluate their sensing and actuating capacities [29]. Besides, actuation of shape memory polymers by electrically resistive heating from nanopaper was achieved by the coating of multi-walled carbon nanotube nanopaper on SMP [30]. Effect on morphological, thermal and mechanical properties was also studied by preparation of a series of graphene oxide filled shape memory polyurethane nanofibers [31]. Although SMPs have been used extensively for several engineering applications, their application in structures has been limited due to their low strength and stiffness. While adding reinforcing fibers did show possibility of improved stiffness in SMP [32,33], the field has been dormant lately. Little effort has been done to explore broadening the structural application of SMPs within the realm of improving their mechanical properties.

This work is focused on further exploring and enhancing the mechanical characteristics of polyurethane based SMP obtained from SMP Technologies Inc., Japan. Baer *et al* [34] first studied the thermomechanical properties of Mitsubishi SMPs (MP3510, MP4510, and MP5510) for potential applications in medical devices like stents or clot extractors. Hu *et al* [35] later studied the effect on shape memory properties of solution type (MS-4510) SMP due to thermomechanical loading. Nishikawa *et al* [36] also studied the effect of short carbon fibers on shape memory properties of SMP (MP5510). Lately, carbon fibers and carbon nanofiber paper (prepared with infiltration method) were incorporated into SMP (MP5510) to increase its electrical conductivity and allow low power high speed electrical actuation [37]. In contrast to these studies, the present study incorporates the use of carbon fiber reinforcing fabric to form a carbon-based SMP composite (SMPC). The paper characterizes the thermo-mechanical properties of SMPC using dynamic mechanical and static tensile testing. The shape retention and recoverability of both SMP and SMPC are also investigated through a series of fold-deploy tests. More importantly, this work investigates the applicability of carbon fabric reinforced SMP by improving its stiffness and strength for potential structural applications.

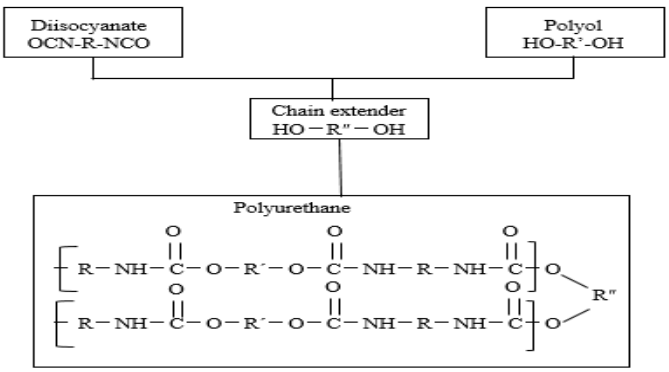



**Fig. 2 Polyurethane based SMP used in the study**

## MATERIAL CHARACTERSITICS

Polyurethane based Shape Memory Polymer (SMP-5510) obtained from SMP Technologies Inc., Japan was used for the entire study. The product is potting type and Table -1 [38] enlists the basic properties of this product.

The material obtained was in the form of two-part system, resin (A) and hardener (B). This SMP family is semi-crystalline with a degree of crystallinity of 3~50 wt. % [39]. The obtained product (SMP5510) was a proprietary product of the company, produced using commonly available diisocyanate, polyol and a chain extender. **Fig. 2** illustrates the chemical formula of the individual components and the final polyurethane product.

**Table -1 Basic Properties of MP-5510**

| Properties | Region | Parameter | Value |
|---|---|---|---|
| Color Tone | - | - | Light yellow |
| A/B Weight Ratio | - | - | 40/60 |
| Viscosity(mPA·s) | - | Solution A | 200-600 |
| | | Solution B | 200-800 |
| Specific Gravity | - | Solution A | 1.062 |
| | | Solution B | 1.215 |
| | | Residue | 1.21 |
| Strength | G/R[a] | Bending Strength (MPa) | 75 |
| | | Bending Modulus (MPa) | 1800 |
| | | Tensile Strength (MPa) | 52 |
| | | Hardness (Shore D) | 80 |
| | R/R[b] | Tensile Strength (MPa) | 20 |
| | | 100 % Modulus (MPa) | 4.5 |
| | | Hardness (Shore D) | 40 |
| Glass Transition Point (˚C) | - | - | 55 |
| Cure | - | Pot Life(Standard) | 180 sec |
| | | Cure Temp(˚C x time) | 70˚C x 1hr-2hr |

[a] G/R , Glassy Region [b] R/R , Rubbery Region

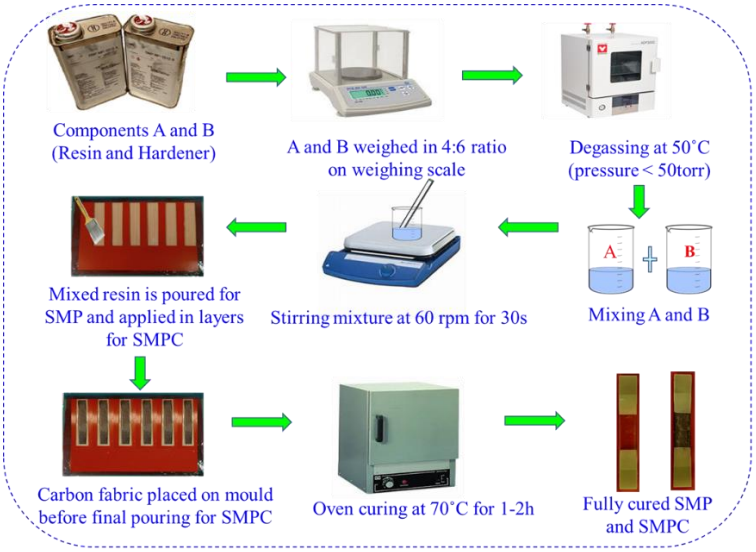



**Fig. 3 Manufacturing Methodology for SMP and SMPC**

## SMP AND SMPC FABRICATION

The fabrication process of the polymers used in the study comprised of four primary steps. The first step is the preparation step, which involves drying of liquids A and B for 1 hour at less than 50 torr pressure. The temperature is fixed at 50˚C while the components are degassed in the degassing oven. In addition, the die is dried for an hour at 70˚C. The second and the most vital step in the preparation of the product is the potting step. Herein, firstly, liquid A and B are placed in vacuum chamber of less than 50 torr. After drying, the component B (the hardener) is filtered using a mesh to remove the thin dried film formed on its surface. This helps achieve a more homogeneous mix. Then, liquids A and B are mixed in 40:60 ratios for 30 seconds with continuous stirring at the speed of 60 rpm. Instantly after the mixing, the mixed solution is poured in the die. The vacuum is discharged immediately as the solution flows completely inside the die. The third and next important step is curing and removal. It involves removal of the die from the vacuum chamber and curing the material for 1-2 hours at 70˚C. Thereafter, finished product is removed

from the die and cured for an additional 1-2 hours at 70˚C, if necessary. The fourth and final step is the cleaning step. The manufacturer recommends methylene chloride for the cleaning.

Preparation of SMPC, for most part remains analogous to SMP. The difference comes after the step when the liquids are mixed. Instead of pouring the complete mixture into the die, it is half-poured, the carbon fabric is placed, coated with a brush of resin to completely infuse resin into the fibre, followed by pouring resin into the remainder of the die mould. It is then placed for curing at 70°C, as quickly as possible, owing to short pot life of the resin (180s). The schematic diagram in **Fig. 3** summarizes the entire process for the manufacturing of SMP and its composite (SMPC).

## THERMO-MECHANICAL CHARACTERIZATION

### Dynamic Mechanical Analysis

Measuring the glass transition temperature ($T_g$) of the SMP used in this study was performed using dynamic mechanical analysis (DMA). The glass transition is associated with the motion of polymeric chains including the neighbouring segments. DMA measures the viscoelastic moduli, storage and loss modulus, damping properties, and tan delta, of materials as they are deformed under a period (sinusoidal) deformation (stress or strain)[40]. For this study, tests were performed using three-point bending mode as this mode is known to work best with stiff samples. The dimensions of the samples used were 45 mm (L) x 12.55 mm (W) x 3.1 mm (T). The temperature range of the test was kept within 25˚C and 75˚C and the loading rate for heating the sample was 1˚C/min while the unloading rate for cooling was 10˚C/min. Oscillation frequency was specified to be 1 Hz. The quantities that were kept in consideration were storage modulus, loss modulus and tanδ. Here, tanδ, the tangent of the phase angle is the ratio of loss modulus to storage modulus. **Fig. 4** shows the setup (PerkinElmer DMA 8000) used for performing the DMA tests.

**Fig. 5** shows the evolution of storage modulus and loss modulus of SMP with respect to temperature. Evidently, the polymer is quite stiff at room temperature and softens as the temperature is raised past $T_g$. The glass transition temperature range is observed to be from 45˚C to 65˚C which is evident from storage modulus and loss modulus function variations against temperature in the graphs presented in **Fig. 5**a and 5b. As the glass transition temperature is defined as the peak of the tan δ curve, by examining the tan δ curve shown in **Fig. 5**c it was concluded that the $T_g$ for the current polyurethane based SMP is approximately 62˚C.

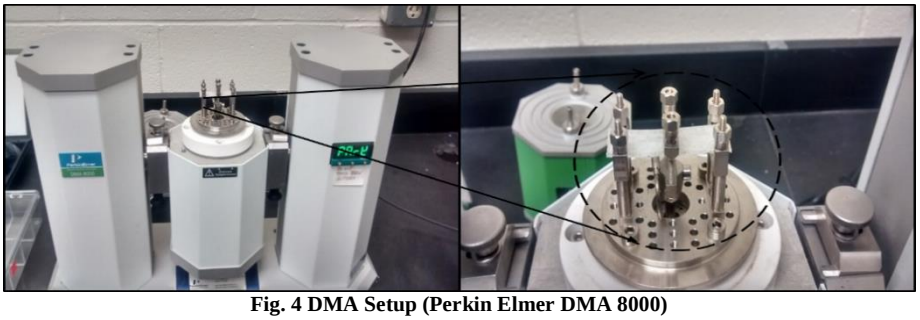
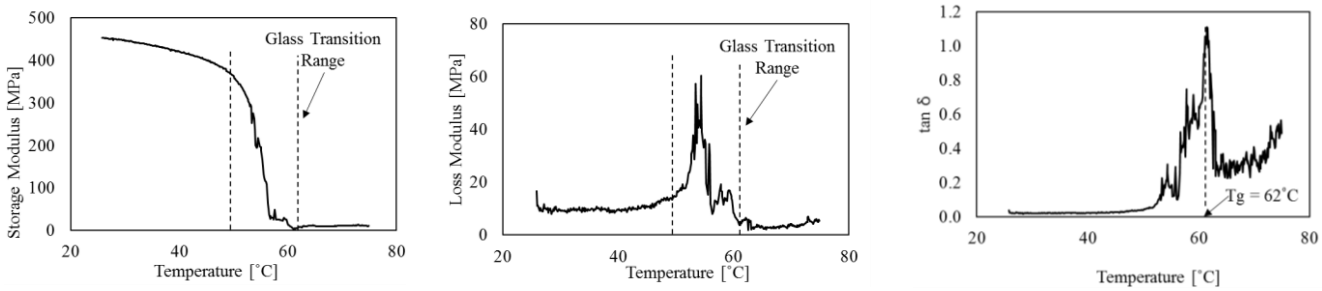


**Fig. 4 DMA Setup (Perkin Elmer DMA 8000)**




**(a)** **(b)** **(c)**

**Fig. 5 Dynamic mechanical analysis results: (a) storage modulus vs. temperature (b) loss modulus vs. temperature (c) tanδ vs. temperature**

## SHAPE MEMORY CHARACTERIZATION

Shape memory retention is the discerning quality of SMPs which differentiates them from other conventional polymers. Hence, it is of utmost importance to characterize SMPs for its shape retention and shape recovery characteristics. In general, universal testing machine is used to test the samples for stress-strain curves. Shape recovery and retention ratios are then calculated from those curves. These traditional tests are not very suitable for all SMP types. For this study, 'fold-deploy' shape memory tests were performed to evaluate shape memory characteristics. The test

essentially consists of three steps: firstly, the samples are heated above the transition temperature using a heat gun. As the material flexes, it is bent about its central axis. The maximum bending angle is recorded at this point and hereafter named as $\theta_{max.}$ In the next step, the bending forces are held constant and the specimen is cooled below its transition temperature. The forces are then removed and the bending angle is recorded again at this stage and is termed as $\theta_{fixed}$. For the third and final step, the specimen is heated in steps until the maximum shape recovery is observed. The bending angle is recorded at intervals of 20s and termed as $\theta_i$. The shape recovery process for pure SMP is shown in Fig. 6.

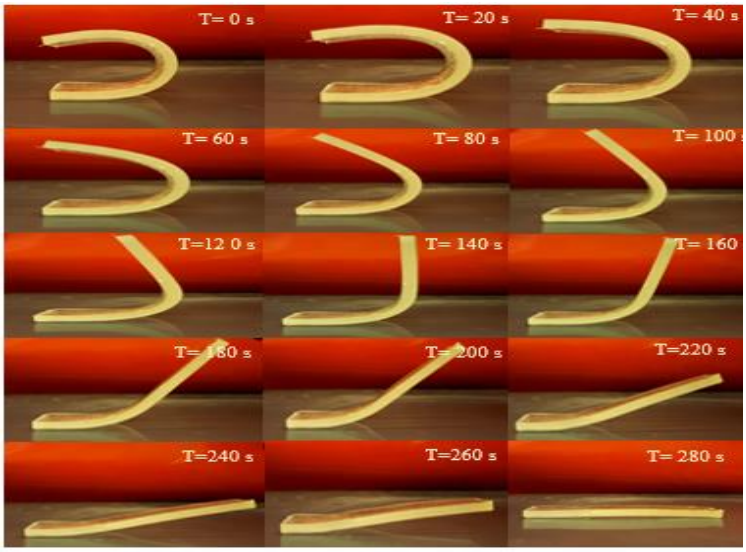



**Fig. 6 Shape recovery in pure SMP**

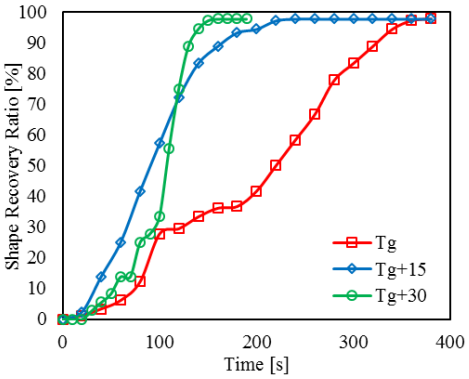



**Fig. 7 Effect of heating temperature on shape recovery ratio**

The shape retention and recovery ratios are calculated as follows:

$$Shape\ Retention = \frac{\theta_{fixed}}{\theta_{max}} \times 100\ \% \quad (1)$$

$$Shape\ Recovery = \frac{\theta_{max} - \theta_i}{\theta_{max}} \times 100\ \% \quad (2)$$

It was found that the polyurethane based SMP used in the study has very high shape retention and shape recovery ratios. Shape retention as high as 99 % was observed for pure SMP samples. Also, in order to investigate the influence of heating temperature on shape recovery ratio, shape recovery performance (for the final step in fold-deploy test) was measured at varying heating temperatures (
Fig. 7). The heating temperatures considered are $T_g$ (62 °C), $T_g$+15 (77 °C) and $T_g$+30 (92 °C). The bending angle in consideration is 180˚. It was observed that for the current SMP product, maximum shape recovery ratio which was 98% was independent of the heating temperature. However, increasing the heating temperature above $T_g$ increased the rate of shape recovery significantly.

## STATIC TENSILE TESTING

In order to mechanically characterize the pure SMP and investigate the impact of adding carbon fiber fabric on the strength, ultimate strain and Young's Modulus of the so formed SMP composite (SMPC), static tensile tests were performed. The quasi-static tests were performed in a displacement controlled mode with a constant crosshead speed of 2 mm/min. A ±20kN load cell was used with the testing machine. An extensometer with a gauge length of 25.4 mm was used at approximately mid height of the coupon specimen to measure axial strains. **Fig. 8** shows the universal tensile testing machine setup for the testing of coupons. As illustrated in Fig. 8, the used specimens had a rectangular shape and were 158.75 mm long, 25.4 mm wide and 12.7 mm thick with a gage length of 44.45 mm. Chamfered tabs with bevel angle of 7-8˚ were used at both ends of the specimen.

In the static tensile tests, engineering stress and strain properties were used to determine the characteristics of SMP and SMPC. Stress-strain plots were generated for pure SMP and SMPC and compared for primarily three parameters, tensile strain at failure, tensile strength and Young's modulus in the linear elastic region. The carbon fabric used in the SMPC was aligned in the longitudinal direction of the specimen. In designing the SMPC specimens, the Young's modulus of the composite ($E_c$) was represented as:

$$E_c = E_m V_m + E_f V_f \quad (3)$$

where, $E_m$ and $E_f$ are the Young's modulus for matrix and fiber, respectively, and $V_m$ and $V_f$ are the volume fibre fraction of matrix and carbon fibre, respectively. The volume fraction of the carbon fibre for the samples tested was approximately 2%. From the bar graph in **Error! Reference source not found.**, it can be observed that with the addition of carbon fibers, the stiffness for SMPC increased considerably from its pure SMP counterpart.

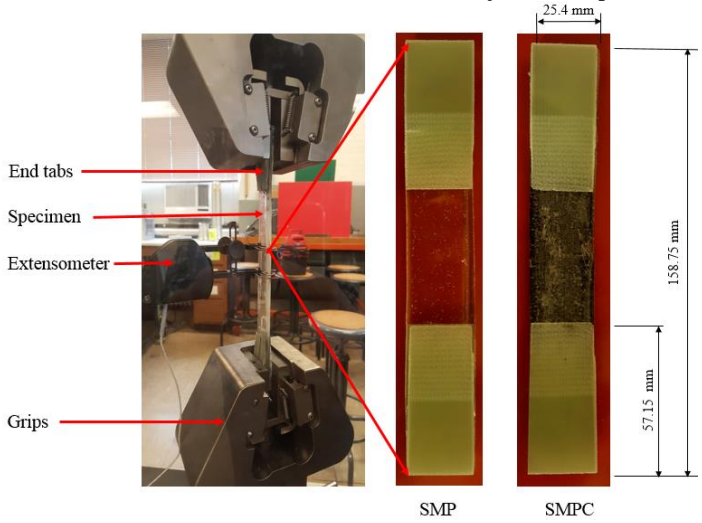



**Fig. 8 Tensile testing setup and specimens**

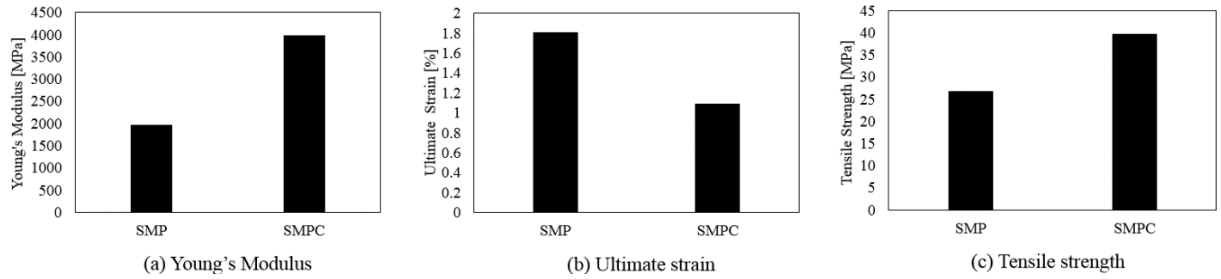



**Fig. 9 Tensile testing results for SMP and SMPC**

The modulus for the SMPC specimen was observed to be 3980.56 MPa, whereas for the SMP it was close to 1977.60 MPa. Stiffness nearly doubled for SMPC specimen, leading to a rise of approximately 100%, as was expected from theoretical calculations based on the rule of mixture for composites as described in Equation 3.

Fracture stress and strain values were also observed from the longitudinal stress-strain curves of the SMP and SMPC. The comparison is elaborated via the bar graph in Fig. 9. Maximum strain observed for the pure SMP samples was close to 1.88 % while for SMPC it was observed to be 1.09 %. This approximately 40% reduction in the ultimate strain value can be attributed to the limiting strain capacity for carbon fibers used as reinforcement in the SMP matrix. The fracture stress increased by approximately 50 % for the SMPC compared to SMP.

**Effect of Deformation**

The developed SMP and its composite (SMPC) have the potential to be used in various structural applications. It is thus of utmost importance that the mechanical properties of the material do not degrade considerably after substantial usage. To account for this, the mechanical properties of SMP and SMPC were examined prior to and after undergoing extreme cycles of deformation and shape recovery. Each cycle consisted of first heating the specimen above $T_g$ to render it flexible. Next, the specimen was subjected to excessive deformation in the form of bending (with a maximum bending angle of 180˚), followed by cooling to lock the strains. Finally, the specimen was heated again past the glass transition temperature to recover back its original shape.

Three cases were considered for this study, namely, no deformation as-built case, one shape memory cycle case, and four shape memory cycles case. The parameters of interest were elastic modulus, strength and ultimate strain. A summary of the results is shown in Fig. 10. As shown in Fig. 10 (a), the stiffness decreases considerably as the sample undergoes 180° shape memory bending cycles, for both SMPC and SMP specimens. Notably, the major drop in stiffness is observed after the first shape memory cycle and the drop after subsequent cycles is very small. For example, in the case of SMPC samples, as the sample undergoes 1 shape memory cycle, the bending stiffness is reduced by 45 % and there is a minor reduction of 5% in stiffness when the sample undergoes 4 shape memory cycles. Also, for all three cases, Young's modulus increased for the case of SMPC in comparison to pure SMP.

However, as the specimen underwent four shape memory cycles, the percentage increase in modulus for SMPC compared to SMP dropped down to merely 18% as opposed to the case when it didn't undergo any shape memory cycles, where the rise was approximately 100%. This may be due to the considerable rearrangement of molecular chains inside the SMP matrix leading to slip between the carbon fibre fabric and the SMP matrix and reduced interlocking of the fibre with the matrix.

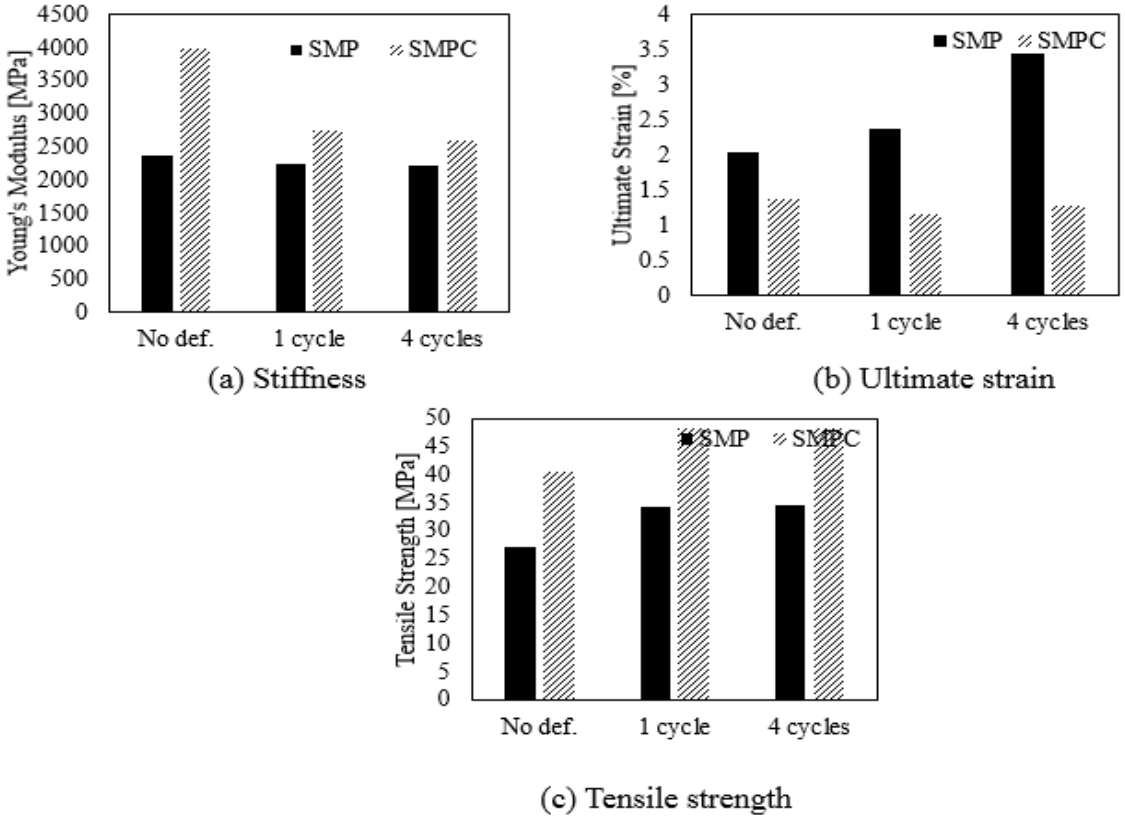



**Fig. 10 Effect of 180˚ bending shape memory cycles on mechanical properties of SMP and SMPC**

On analysing ultimate strain of the samples (Fig. 10 (b)), it was observed that the strain of SMP increased with increasing the number of cycles by as much as 69% for the 4 cycle case when compared with as built case. This increase may be attributed to the Brownian motion and changes in molecular arrangement in the SMP matrix, which lead the specimen to become excessively flexible after going a number of characteristic shape memory cycles. On the other hand, the number of cycles seem to have opposite impact on the SMPC, where a decrease of 16 % was noted in the ultimate strain when SMPC was subjected to one cycle only, when compared with no deformation as-built case. Subjecting the SMPC to more cycles didn't seem to have significant additional impact on the ultimate strain as it only resulted in a decrease of 1.15 % compared to the one cycle case. This observation with major deviation after 1 cycle and no considerable change after subsequent cycles, in ultimate strain holds true even for stiffness as evident in Fig (12(a)) and even for strength (Fig 12(c)) as described further.

Analysing the tensile strength of SMP and SMPC (Fig. 10 (c)) revealed that, for the as-built case, the tensile strength of SMPC is higher than that of SMP by approximately 48 %. This difference is analogous to the results previously obtained (see Fig. 10(c)). Also, on average, this rise in stiffness comparing as-built SMPC and SMP specimens, is maintained as the samples undergo one characteristic shape memory cycle. The rise between SMP and SMPC is not as significant (39 % rise) as the sample undergoes 4 shape memory cycles. This may be due to flexing of samples with large number of cycles. In addition, comparing only SMP samples, the tensile strength rises by 25 % as the sample undergoes one shape memory cycle and the rise is approximately 1-2 % as it further undergoes 4 characteristic shape memory cycles. Analogous to this, comparing only SMPC specimens, the tensile strength first rises by approximately 18 % for one cycle and drops to 1-2 % as it further experiences more shape memory cycles. The reason for this behaviour can be attributed to the fact that the deformation and failure govern the first cycle, whereas training and memorizing effect of the shape memory polymer governs the subsequent cycles, causing it to stabilize after a while.

**Effect of Bending Angle**

A parametric study was conducted with the SMP and SMPC specimens to study the effect of deformation angle on the mechanical properties of the polymer in pure and composite forms. Three different bending angles, namely 45˚, 90˚, and 135˚ were considered in the study. Initially, specimens (both SMP and SMPC) were heated above their glass transition temperature and deformed to required angles. Subsequently when the specimens were cooled down the applied bending force was removed and strains were kept fixed. The state was maintained for a duration of 1 hr and then the samples were heated again past their transition points to recover back the locked deformation. Fig. 11 shows specimens under various levels of deformation. The prepared samples were then tested to failure in MTS testing machine to obtain their characteristic mechanical properties as shown in Fig. 12.

The bar graph in Fig. 12 (a) shows the variation of Young's modulus for SMP and SMPC after being deformed under various deformation levels. Quite evidently, the elastic modulus for both SMP and SMPC samples decreased as the deformation angle increased. However, the rate of decrease in stiffness had small dependency on the angle of deformation. The major decline in stiffness (16.58 % for SMP) and (29.87 % for SMPC) occurred after first bending angle deformation of 45°. Besides, analysing the case with no deformation and the case with 135˚ bending angle deformation, for pure SMP, the percentage decrease in the modulus was calculated to be 36.56% whereas for SMPC the decrease was 45.62%. Furthermore, as expected, the modulus of elasticity for the SMPC was always higher than that of pure SMP. Comparing SMP and SMPC, the maximum increase in stiffness was observed for the samples with no deformation at 67.18% while for the case of 135˚ deformation, the rise was 53.35%. Evidently, as the SMP/SMPC undergoes any level of deformation, the macromolecular chains experience high disorientation and specimen becomes quite flexible, leading to a stiffness decline.

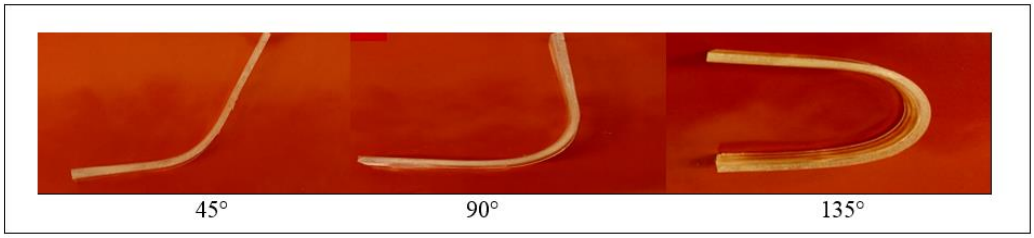



**Fig. 11 SMP deformed under different angles of deformation**

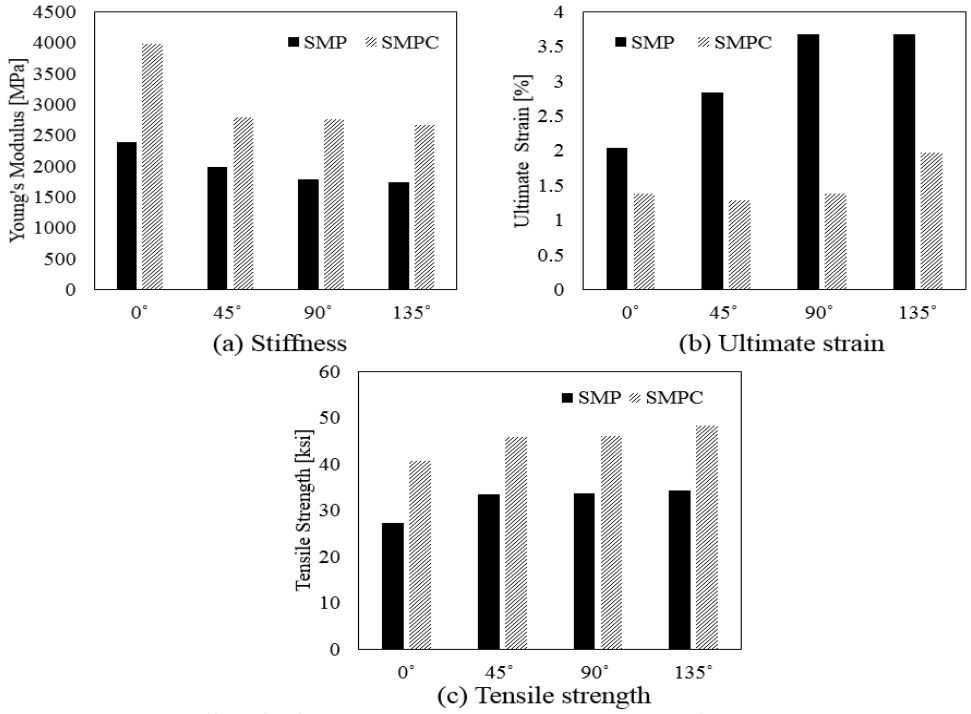



**Fig. 12 Effect of deformation angle on mechanical properties of SMP and SMPC**

For the case of tensile strain, as the Brownian motion of the specimen increases, the sample becomes flexible and increase in the strain carrying capacity is observed. This may be attributed also to the slackening effect in intermolecular chains found in polyurethane based shape memory polymer products. The tensile strain at failure, comparing specimens with no deformation and samples with 135˚ deformation, the rise was observed to be 44.4% and 30.22% for SMP and SMPC cases, respectively. The maximum strain observed was 3.68% and 1.97% for the SMP and SMPC, respectively, for the 135˚ bending angle deformation case. The ultimate strain for the SMPC samples is lower

than that of the SMP, for all four deformation levels due to the limiting strain carrying capacity of carbon fiber fabric used in manufacturing SMPC.

From the results obtained for tensile strength, as depicted in Fig. 12 (c), SMPC specimens at all levels of deformation have strengths higher than their SMP counterparts. Comparing SMP and SMPC samples, the maximum rise of 48 % is for samples with no deformation. However, with the increasing degree of deformation, the percentage rise between SMP and SMPC decreases, like for the 135° bending case, the difference in tensile strength between SMPC and SMP is merely 40%. When individually comparing the SMP and SMPC specimens, the tensile strength increases compared to the as-built case (22 % and 13 % for SMP and SMPC, respectively) with 45° bending angle deformation. The value stabilizes after that and no substantial increase is observed with further deformation of 90° and 135° for both SMP and SMPC. Comparing no deformation case and 135° bending case, the tensile strength increases by 25% for SMP case and 19 % for SMPC case, respectively.

## CONCLUSIONS

In this work, SMP and its carbon fibre composite (SMPC) were manufactured and characterized for their shape memory and thermo-mechanical characteristics. Few highlights of the research study can be concluded as follows:

- The $T_g$ of the SMP was found to be 62˚C. Excellent shape recovery ratio (98 %) and shape retention ratio (99 %) were also observed. Also, increasing the heating temperature above the $T_g$, increased the rate of shape recovery significantly.
- Stiffness nearly doubled for SMPC compared to SMP. Fracture strain decreased for SMPC samples by approximately 40 % owing to the controlling strain carrying capacity of the carbon fiber.
- Stiffness and tensile strength decreased for both SMP and SMPC with the increase in shape memory cycles. Notably, the major decline occurred after the first cycle. Besides, the ultimate strain of SMP increased by as much as 69% after undergoing 4 shape memory cycles compared to as-built case. Contradictorily, for SMPC, ultimate strain declined by approximately 17% for the same case scenario.
- With the rise of degree/angle of deformation, comparing as-built and 135° deformation cases, 36.56% decline in stiffness was observed for SMP, whereas for SMPC the decrease was 45.62%. Additionally, with the rise of degree of deformation, the average rise in tensile strength between SMP and SMPC decreased to roughly 40% for 135° case compared to 48% for the no deformation case.

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