Influence of Silicon Carbide Nano Fillers on the Thermal, Mechanical and Shape Memory Properties of Tpi Shape Memory Polymer Composites

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Abstract

TPI, also known as artificial eucommia rubber, is a thermo-responsive SMP created by molecular recombination and modification with contemporary polymer synthesis and modification techniques. In this paper, TPI shape memory polymer is reinforced with different weight proportions (0, 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 %) of Silicon carbide nano particles (SiC) and specimens were fabricated with the aim of enhancing the properties of neat TPI polymer composites. Some thermo-mechanical characteristics were investigated and interpreted, including the differential scanning calorimeter (DSC) test, dynamic mechanical analysis (DMA) test, thermal conductivity test, compression test, and a few shape memory properties such as shape recovery ratio and shape recovery rate. TPI shape memory polymers exhibit strong shape memory capabilities and the best mechanical properties based on the testing results for the specimen TPI with 0.8 % SiC weight fraction.

1. Introduction

Shape Memory Materials are materials with the unusual capacity to restore their permanent forms after being exposed to external stimuli such as heat, electricity, wetness, and light [1]. Shape memory polymers (SMP), also known as smart polymers or stimuli-responsive polymers, are part of a larger class of materials known as Shape Changing Polymers (SCPs) [2-3]. The ability to specifically alter material characteristics for individual applications is a key benefit of SMPs, and SMPs do not require costly or difficult production techniques [4]. As a result, shape memory polymer composites have a wide range of applications and will play an essential role in the development of future sensors, aircraft, textiles, biomedicine, micro-robots, 3D printing, and other sectors [5-9]. Trans-1, 4-polyisoprene (TPI) is an isomer of cis-1, 4-polyisoprene (NR). TPI has gotten a lot of interest because of its unique properties, such as its readily customized transition temperature (Tt) at 317-325 K [10].

Many features of pure SMPs / TPIs render them inappropriate for different applications, particularly those requiring strong mechanical qualities like as stiffness and strength, as well as high recovery force, excellent electrical conductivity, and self-healing capabilities. As a result, recent research on shape memory polymers has moved focus to the development of shape memory polymer composites in order to examine in more depth their mechanical and other thermo-mechanical properties in order to fulfil as required criteria in a variety of application areas [11-12]. Superior mechanical qualities have been observed by many researchers over the last few years due to the addition of carbon nanotubes (CNTs) to all of the existing filler materials. According to the findings of Qing-Qing Ni et al.'s study on the shape memory effect and mechanical characteristics of CNT SMP nano-composites, adding a tiny quantity of CNT fillers to the SMP matrix improved mechanical parameters such as modulus [13]. Jianxin Teng et al. fabricated a composite by introducing low-cost high density polyethylene (HDPE) into trans-1 4-polyisoprene (TPI) matrix as reinforcing phase. The findings of the research were crystallinity and crystallization temperature increases first and then decreases with the decrease of TPI content, and the maximum value appears when the TPI content is 80%. The shape recovery process of TPI/HDPE hybrid SMPCs is a stepped recovery and the static mechanical properties of TPI/HDPE hybrid SMPCs have

temperature dependence [14]. Guo et al. investigated the TPI-SMP composites with chopped carbon fiber weight fraction of 5 %, 7 %, 9 %, 1 1% and 13 % and reported that the maximum stresses increase rapidly with the number of cycles increasing in the initial phase, and then they approach to a relatively stable value under a constant strain cyclic loading. The experimental results also showed that the TPI with 7 % carbon fiber weight fraction appears to perform best in all the tests [15]. Some new nano materials have been introduced by few researchers [16-20] as a filler materials for the fabrication of TPI composites and they have also proved to be more effective in terms of thermal, mechanical and shape memory properties.

SiC usually act as reinforcement phases to improve the property of a polymer-matrix due to their exceptional mechanical properties, low weight, high aspect ratio, high elastic modulus, chemical stability, and high thermal shock resistance. Hence, in this present work TPI shape memory polymer is reinforced with different weight proportions (0, 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 %) of silicon carbide (SiC) and specimens were fabricated with the aim of enhancing the properties of neat TPI polymer composites. And few thermo-mechanical characteristics are to be investigated, including the differential scanning calorimeter (DSC) test, dynamic mechanical analysis (DMA) test, thermal conductivity test, compression test, and a few shape memory properties such as shape recovery ratio and shape recovery rate. Then the obtained results are to be interpreted accordingly.

2. Materials And Methods

2.1 Materials & Preparation

Trans-1,4-polyisoprene (TPI), acquired from Sigma-Aldrich in India, was the primary matrix raw material employed in this work. Silicon carbide (SiC) were produced and obtained from Quantum Materials Corporation in India as the chosen reinforcement. The surface modification of SiC is then carried out by putting 0.5 grammes of SiCs in 80 ml combination of H_2SO_4/HNO_3 (3:1). The compound mixture was then sonicated numerous times, and the resulting filter was rinsed multiple times with distilled water before being dried at 80°C in vacuum for 8 hours. The reinforcement quantity employed was (0, 0.2 %, 0.4 %, 0.6 %, 0.8 %, and 1 %), and the measured reinforcement was combined with the matrix material before being processed using a high-temperature open mill at 90°C for roughly 10 minutes using a conventional mixing sequence. The TPI was then vulcanised at a low temperature using a sulphur vulcanization technique. The TPI/SiC specimens were then removed and dried at room temperature, and all testing specimens were made using a laser cutting machine.

2.2 Characterization Techniques

The melting and crystallisation transition properties of TPI/SiC hybrid SMPCs with varied mix ratios were measured using a differential scanning calorimeter (Perkin-Elmer Diamond DSC). DSC was primarily utilised to determine the glass transition temperature and the presence of crystallinity in the samples. A Perkin Elmer 8000 dynamic mechanical analyzer was used to measure the dynamic mechanical characteristics. DMA evaluates materials' viscoelastic moduli, storage and loss moduli, damping

characteristics, and tan delta as they are distorted over time. Specimens were scanned at a heating rate of 3 K/min with an oscillation frequency of 1 Hz. The glass transition temperature (Tg) of a material is the temperature at which it transitions from a glassy to a rubbery phase.

The thermal conductivity of TPI/ SiC composite specimens was measured using TA equipment at a set of temperatures. After equally spraying graphite onto specimens with parallel upper and lower surfaces, the upper surface was subjected to a specific laser pulse wave while the bottom surface's temperature response was measured and recorded. The compressive tests were carried out using the BOSE Electro-Force load frame equipment using a force-controlled test. TPI/SiC specimens were cut into small cylinders and evaluated at room temperature in force-control mode with a compressive force of up to 2000 N applied at a rate of 2 N/sec. Shape memory retention is a distinguishing feature of SMPs that sets them apart from other typical polymers. TPI/SiC SMPC shape memory capabilities were investigated using the standard fold/unfold approach. The test procedure consists of the following steps. (i) The specimen was heated to a temperature greater than the transition temperature of TPI and SiC, and at that temperature, the specimen was bent into a U-shape. Its maximum bending angle θm was measured and reported at that time. (ii) The bending forces applied are retained constant, and the specimen is cooled below its transition temperature. The pressures are then released, and the bending angle is recorded once more, this time as θf. (iii) At regular intervals, the specimen is warmed, and the bending angle θi is measured. The shape retention and shape recovery ratios are then computed.

$$Shape\ Retention\ Ratio: R_{SRN} = \frac{\theta_f}{\theta_m} \text{ X } 100\ \%$$

$$Shape\ Recovery\ Ratio: R_{SRY} = \frac{\theta_m - \theta_i}{\theta_m} \text{ X } 100\ \%$$

3. Results & Discussions

3.1 DSC

Figure 1 depicts the DSC thermo gram of carbon nanotube-reinforced TPI shape memory polymer composites at various mass fractions.

From the figure, it is observed that the thermo-gram of TPI shows a peak value at temperature of 317 K, which specifies the transition of state inside the specimen at that value. The number 317 K is also used as the crystallization melting temperature for TPI-SMP composite specimens, and the addition of SiC shifts this value to higher values. When the mass percentages of SiC are raised, the crystallization melting temperature increases somewhat when compared to TPI. This demonstrates that SiC reinforced TPI composites have much higher heat resistance than TPI composites. Furthermore, the crystallization melting temperature indicates an improvement in the micro-phase separation degree of polymeric chains, which becomes a measure of the composites' mechanical qualities. As a result, it is possible to conclude

that increasing the crystallization melting temperature results in good mechanical characteristics of composites.

3.2 DMA

Figure 2 depicts the storage modulus and loss tangent (tan δ) curves generated from the dynamic mechanical study. It can be seen from the figure 2a, the storage modulus of TPI composites are found increased by adding the various weight percentages of SiC reinforcement. Among all the specimens fabricated, the TPI-0.8 SiC composite had shown a highest storage modulus and this is because the inclusion of silicon carbide which enhances the stiffness of TPI/ SiC - SMPC by developing the interfacial stress transmission [21]. Also, at the same time, it is inferred that when the SiC loading increased to 1.0%, the storage modulus is found decreased and this may be occurred due to formation of agglomeration of SiC due to vander waals force of attraction, resulting in a poor interaction between the TPI matrix and the SiC, the stress transfer is reduced. Figure 2b, depicts the tan δ curves of all composite specimens. The glass transition temperature (Tg) of TPI composite may be shown to be 313 K. This value then moves to a lower temperatures, when the TPI is reinforced with different weight percentages of SiC, which is an indication of enhancement of motion between the TPI polymer and the SiC filler.

3.3 Thermal Conductivity

Figure 3 depicts the thermal conductivity of TPI/ SiC shape memory polymer composites tested at 303 K, 323 K, and 343 K. It is observed, the thermal conductivity of TPI composites increased with the increasing content of filler loading up to 0.8 wt. %. Because SiCs have a high thermal conductivity, they create a continuous thermal conduction network chain in the TPI polymer. As the SiC wt. % increases, the SiC filler becomes more densely packed, strengthening the heat-flow route [22]. At the same time, as the SiC loading increases above 0.8 wt.%, the composite's improved range of thermal conductivity decreases. The explanation might be the creation of aggregation bundles of SiC, which causes poor adhesion with the matrix phase and so contributes less to the heat-conductive network chain.

3.4 Fracture Stress

Figure 4 describes the effect of different filler loading on fracture stress. It has been observed, the fracture stress shows an increasing trend towards the addition of increasing weight percentages of SiC loading as a filler material. This might be because the molecular chains of SiC and the TPI matrix re-form a uniform distribution after adding an adequate amount of SiC with higher mechanical characteristics. Because the reconstructed two-phase molecular segments have higher tensile strength, the specimens' TPI fracture stress rises. The fracture stress becomes low for the TPI specimen loaded with 1.0 wt.% SiC, which occurs when the two-phase molecular chains entangle and the molecular organisation inside the material is disordered after adding excessive SiC.

Furthermore, as indicated by the plots, the fracture stress of all specimens is temperature sensitive, and as the test temperature rises, the fracture stress of all specimens decreases accordingly. When the test

temperature is 303 K, the fracture stress of all specimens is greater than 8 MPa, but it is greater than 4.5 MPa when the test temperature is raised to 343 K. This is due to the fact that TPI approaches the transition temperature as the test temperature rises. When the crystalline component of TPI begins to melt, the kinetic energy of the molecular segment rises. The mechanical properties of TPIs are diminished as a result of the crystallization being destroyed.

3.5 Shape Memory Properties

The TPI-0.8 SiC specimen is heated to a temperature 325 K above Tg, then cooled, and the curve is plotted at regular time intervals as shown in figure 5. The specimen, on the other hand, rebounds more quickly, with a fast recovery of more than 70 % of the bending detected during the first minute, although the shape recovery rate steadily declined subsequently. The bending angle curve and the shape recovery ratio curve intersect at a time span of roughly 60 seconds. This is because the reversible phase of TPI in the pre-deformation process is stretched from an ordered arrangement to a coiled condition under external strain [23]. In 6 to 7 minutes, almost 90% of the deformation was restored. Furthermore, the bending angle hits 90° relatively quickly, i.e. during the first minute, and then progresses slowly after that.

After 10 minutes, the bending angle is 7° and the form recovery rate is 96.5 %. The accumulated energy in frozen molecular chains is progressively released, and the thermal motion of molecules in TPI is similarly accelerated. As the weight percentage of SiC grew, the form recovery rates of all specimens decreased. This might be because the TPI/ SiC composite structure in a polymer composite has a higher recovery force than a TPI matrix. A SiC -loaded specimen is bigger than a TPI specimen and takes more energy to achieve form restitution. When the temperature remains constant, form memory recovery takes longer, resulting in a decrease in shape recovery rate.

4. Conclusions

Thus, the TPI shape memory polymer was reinforced with different weight proportions (0, 0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 %) of silicon carbide and the specimens were subjected to mechanical, thermal and shape memory test according to their standards and the results were interpreted. The main conclusions drawn from this study are:

- The Tg of the TPI an TPI/ SiC composite specimens were measured from the peak of the tan δ curve.

- The crystallization melting temperature from the DSC curve for TPI/ SiC composite specimens gets increased marginally when compared to TPI.

- Among all the specimens fabricated, the TPI-0.8 SiC composite had shown a highest storage modulus and this becomes a good indication of better mechanical properties.

- The thermal conductivity of TPI composites increased with the increasing content of filler loading up to 0.8 wt. % because of straight heat-flow path.

- The fracture stress of all specimens is temperature dependent, and as the test temperature rises, so does the fracture stress of all specimens.

- More than 70 % of the bending was regained during the first minute of the form memory test.

- The specimen possess excellent shape memory recovery property with almost 97.4 % of recovery ratio and the shape recovery rates of all the specimens decreased with increase in weight percentage of SiC.

Declarations

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Conflict of Interest The authors declare no confict of interest

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Figures

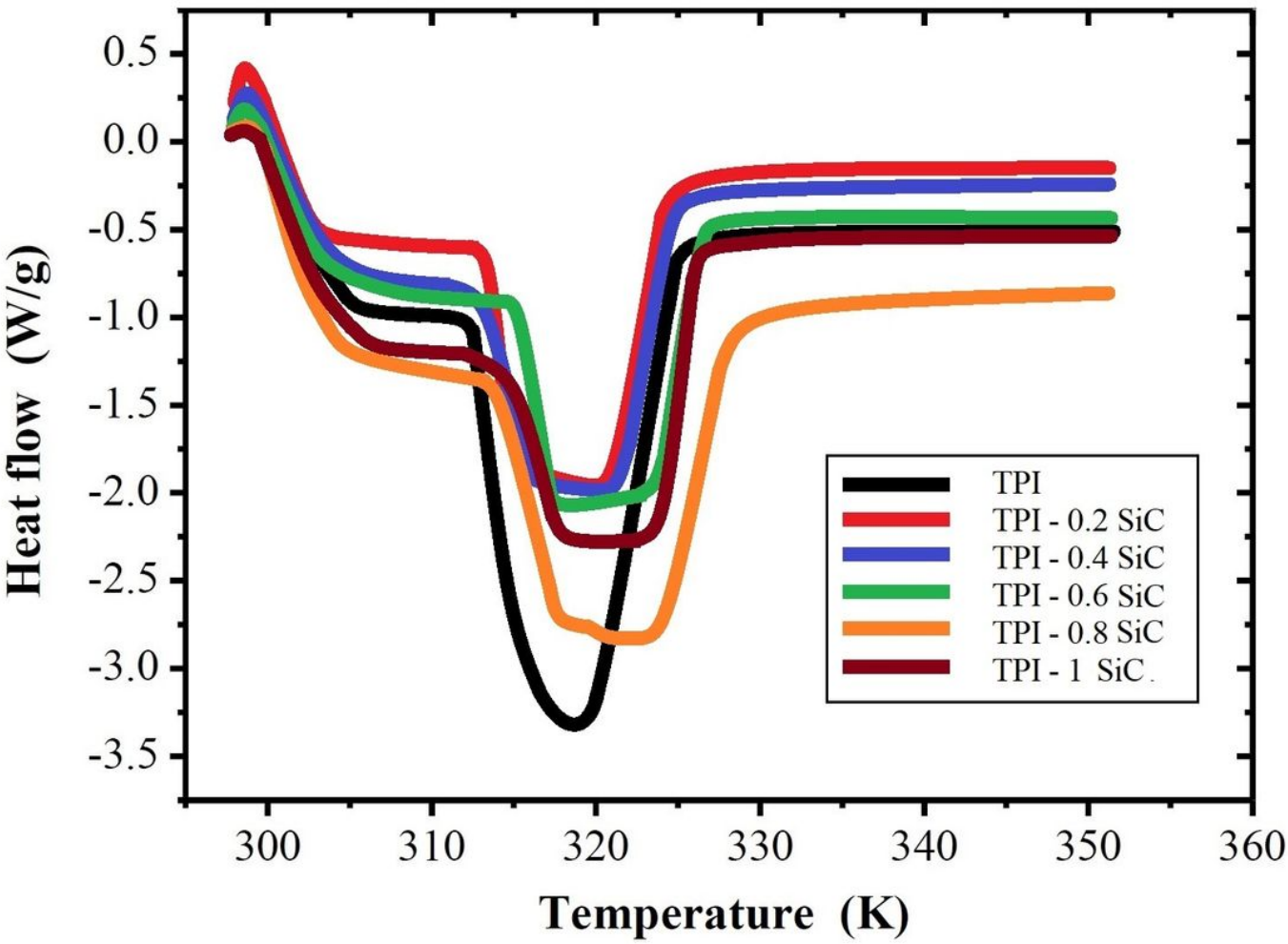

Figure 1

DSC thermo gram of carbon nanotube-reinforced TPI shape memory polymer composites at various mass fractions

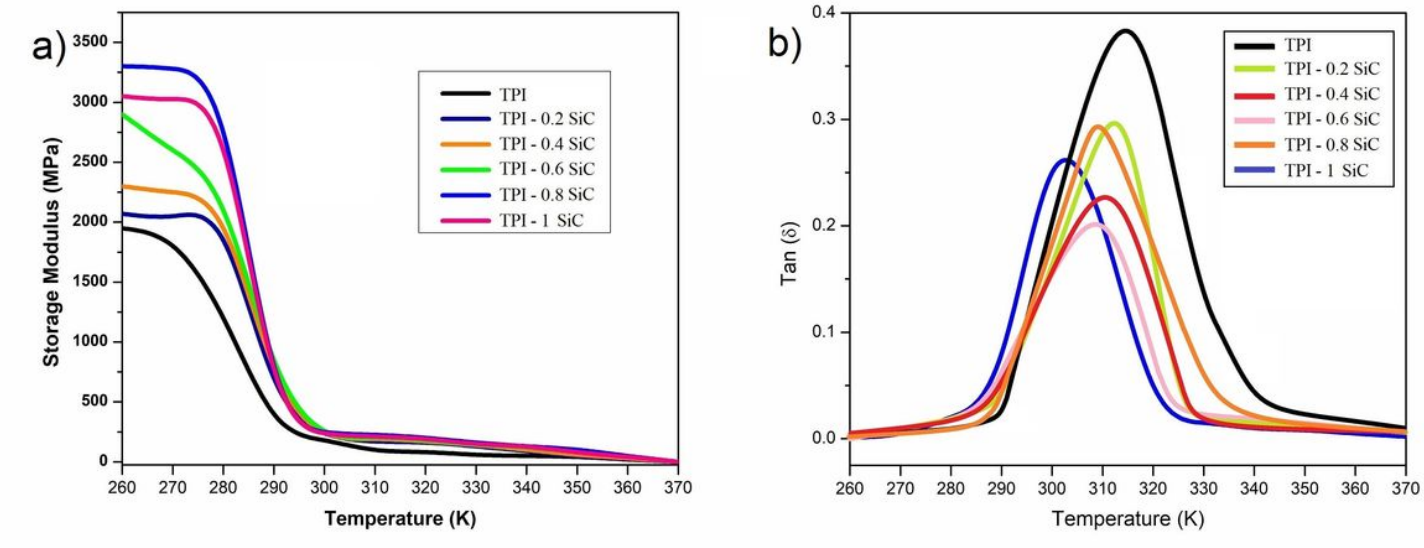

Figure 2

DMA curves of TPI/ SiC *Shape memory polymer composites with different weight percentages of* SiC *(a) Storage Modulus (b) Loss Tangent Curve*

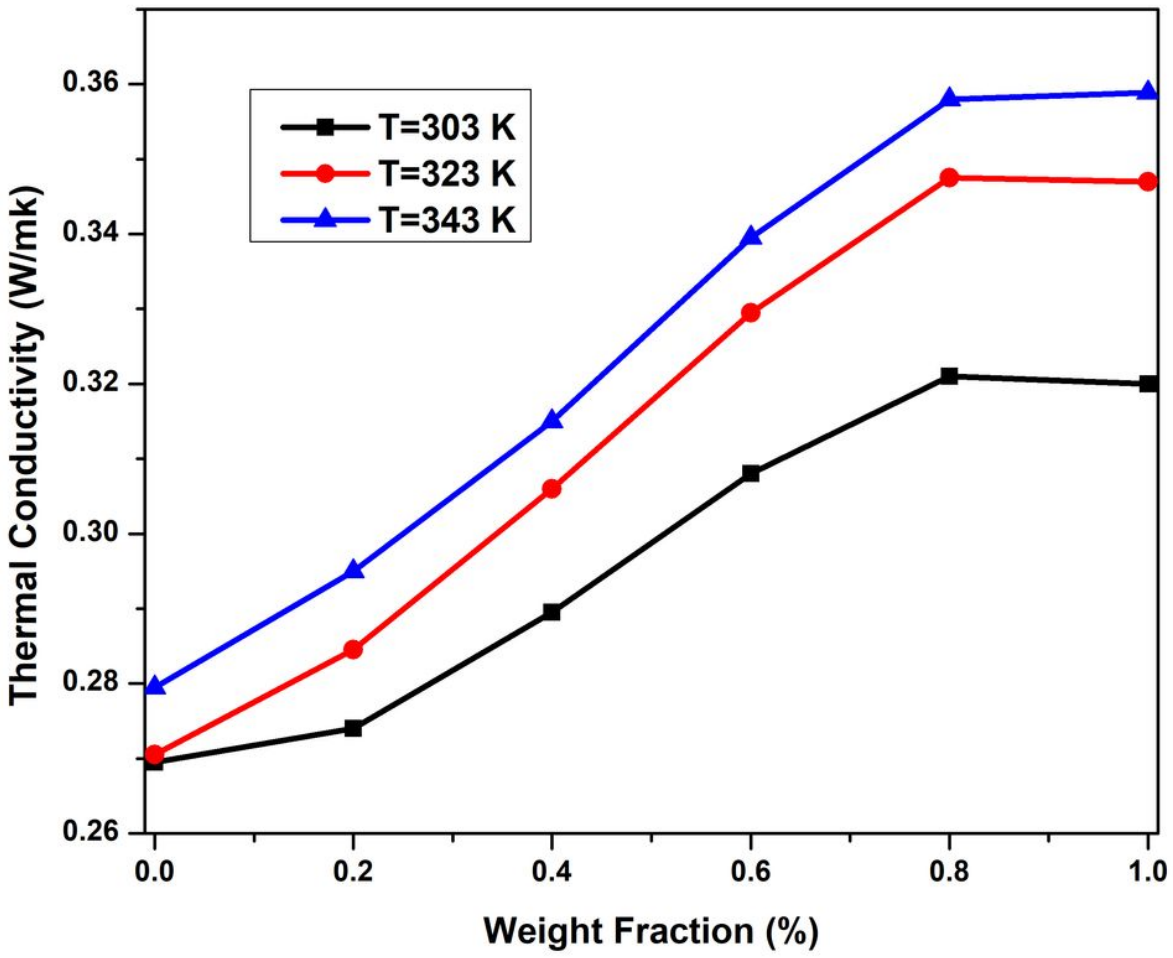

Figure 3

Thermal conductivity of TPI/ SiC shape memory polymer composites

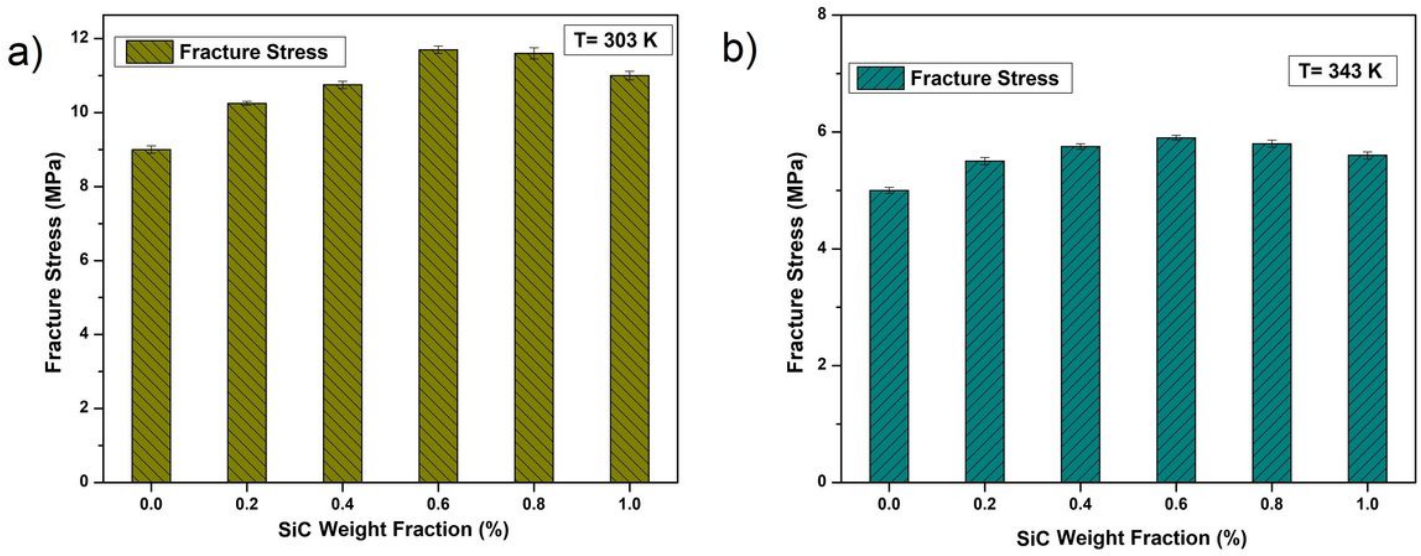

Figure 4

Fracture Stress for the various weight percentages of TPI/ SiC *Composites*

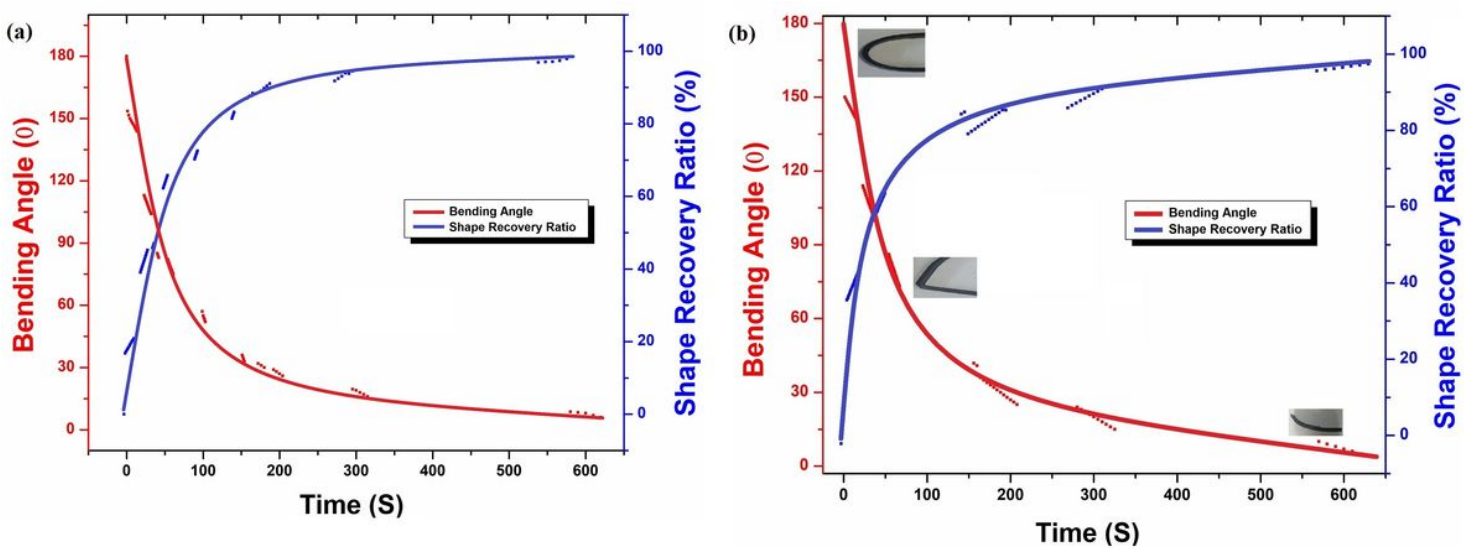

Figure 5

Shape Recovery Curve of TPI – 0.8 SiC Specimen