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Synthesis and Analysis of Shape Memory Effect in Shape Memory Polymer

[1] K.B. Arun, [2] U.S. Mallik, [3] M. K. Ranganath
[1] M.Tech. scholar, Department of Mechanical Engineering, SIT, Tumakuru, Karnataka, India.
[2] Professor, Department of Mechanical Engineering, SIT, Tumakuru, Karnataka, India.
[3] Asst. Prof. Department of Mechanical Engineering, Jain University, Banglore, Karnataka, India

Abstract: **Shape memory polymers (SMP's) are materials capable of recovering their permanent shape from a temporary one upon the exposure to a specific stimulus, such as heat, stress, magnetic field. The shape memory effect in polymers comes from their unique molecular structures. If only small loads are needed then shape memory polymers offer an attractive alternative to shape memory alloys due to their low manufacture cost and ease of processing. The main purpose of this project is to synthesize shape memory polymer using polyethylene glycol (PEG-6000) as the soft segment and isophorone diisocyanate (IPDI) as the hard segment and to analyze its shape memory effect. Additional fillers such as CNT and Graphite can be used during synthesis to enhance rigidity and strength of shape memory polymer.**

Keywords: Shape memory polymer, Shape memory effect, Soft segment, Hard segment.

I. INTRODUCTION

Shape memory polymers (SMP) are the class of material which will respond to external stimulus. SMP can remember a secondary shape and it can return to its permanent shape when external stimulus is applied. External stimulus can be heat or stress. Shape memory effect (SME) in SMP can be defined as the ability of the material to return to its original shape from secondary shape. Shape memory effect is not an intrinsic material's property it can be designed and altered by changing a polymer's structure, morphology, and the various processing parameters during fabrication and programming. In this study one-way thermally responsive SMPs are studied, and the recovery process is irreversible. The transition temperature (Ttrans) this is the temperature at which a SMP recovers to its permanent shape and it is consider as an impartment parameter [1].

Shape-memory polymers can be used in many applications, such as deployable space structures, space curable composites, inflatable tubes, heat shrink tubes, breathable textiles, medical supplies, choke device for auto motives, actuators and sensors, stents, insulators, and packaging materials. Thermoset- and thermoplastic type

Shape memory polymers can be used in self-healing synthetic leather and paint [2].

Shape-memory alloys (SMA) have been studied by various people for over 40 years, hence SMAs finds variety of applications including such as eye-glass frames, self-adjusting orthodontic wires, stents, actuators, and temperature-responsive valves. The cost involved in manufacturing SMA is more when compared with SMP. SMP show shape-memory behavior, and they are the best and a cheaper alternative due to their ease of preparation and processing. Shape recovery in alloys is enthalpy-driven, and shape recovery in polymer is an entropy-driven process. SMP shows greater deformations up to several 100% in certain cases when compared with SMA. Polymer networks are less susceptible to creep [3].

Carbon nanotubes can be used as fillers to improve mechanical and electrical properties of SMP [4].

Mechanical properties of SMAs are significantly limited, and the toxicity levels in such alloys are disadvantageous for biomedical applications. In addition SMAs are not degradable, and they require an additional removal operation after implantation of any material inside the human body. But when SMP is compared with SMA it has many advantages such as various chemical structures, high shape-stability, low recovery temperature, easy processing, and lightness. Shape memory effect in SMP depends on the nature of the polymer structure. Many shape-memory polymers have been prepared, and their shape-memory effects have been studied. Among these polymers, some polymers are biodegradable and it has several potential biomedical applications. For medical applications SMP must have high shape-recoverable property, and the recovery temperature should be near the body temperature [5].

It has been studied that thermoplastic SMPs exhibit relatively large elongations but poor shape recovery, while thermoset SMPs have excellent shape fixity and recovery properties [6].

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In the present study shape memory polymer was prepared using polyethylene glycol (PEG-6000) as the soft segment (58%) and isophorone diisocyanate (IPDI), 4,4'-Methylenebis (phenyl isocyanate) (MDI) as the hard segment (together 42%). Some trail experiments was carried out and shape memory effect test was conducted.

II. EXPERIMENTAL WORK

2.1 Set up

1. 50ml round bottom three neck flask.
2. A silicon oil bath with a temperature controller equipped with magnetic or mechanical stirrer.
3. A thermometer to monitor temperature.
4. The flask plugged with continuous flow of dry nitrogen.
5. Vacuum furnace.

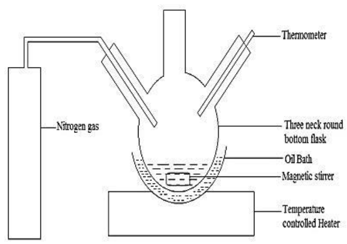

Fig. 2.1 Set up required to prepare SMP

2.2 Raw materials

1. Polyethylene glycol 6000 (PEG-6000) soft segment.
2. N, N- dimethyl formamide (DMF) solvent.
3. Isophorone diisocyanate (IPDI) hard segment.
4. Dibutyltin dilaurate (DBDT) catalyst.
5. Polyethylene glycol 200 (PEG-200) cross linking agent.
6. 4,4'- Methylenebis (phenyl isocyanate) (MDI) hard segment.
7. 1,4- Butanediol (BDO) chain extender.

2.3 Procedure

1. First the raw materials required for synthesis are determined. The raw material consists of soft and hard segments. Soft segment is taken 58 w% and hard segment is 42w%. Soft segment consists of PEG 6000 and hard segment consists of IPDI and MDI.
2. Measured amount of PEG-6000 + DMF + IPDI + DBDT catalyst are added into the bottom round three neck flask.
3. Above chemicals are reacted for 2 hours at 900C.
4. After 2 hours of reaction at 900C PEG-200 + MDI are added into the flask.
5. The above mixture is allowed to react for 1 hour at 900C.
6. After 1 hour BDO is added drop wise at 600C and the reaction is continued for 1 hour.
7. Then the mixture is poured in glass mould and the mixture is baked at 600C for 12 hours, 800C for 24 hours and at 1000C for 8 hours respectively in a vacuum oven.
8. Then the sample is removed from the mould and further tests are carried out.

2.4 Shape memory effect

The Shape Memory Effect was determined using bend test for all 5 SMP samples with dimensions of 100x30x1mm3(length X width X thickness) at room temperature. The specimens were bent into a U-shape around a mandrel of known diameter (d=30 mm) at room temperature. To measuresthe angle the projections of the samples were drawnson the paper and thesangle was thensmeasured with theshelp of asprotractor. The % of SME is measured by SME = θm/180º-θe Where, θm = angle recovered on heating. θe = angle recovered on unloading. The recovery ratio is measured by RN = θ0-θN/ θ0 where θ0 = initial angle before heating and θN = final angle after heating. Below figures shows the shape memory effect in SMP which was used in present work.

Fig. 2.2 SMP sample before loading i.e. original position

Fig. 2.3 SMP sample after un-loading

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Fig. 2.4 SMP sample after heating exhibits shape memory effect

2.5 Glass transition temperature

Glass transition temperature can be defined as the temperature at which a change in physical properties occurs in a substance, such as change in phase or change in crystalline structure. At this temperature substance may acquires or loses a distinctive property. In the present work glass transition temperature was characterized using Differential Scanning Calorimeter (DSC). DSC is a technique where glass transition temperature is determined by plotting the heat flow on Y axis and temperature on X axis. DSC for present work was carried out in temperature range of 0 to 1000C.

2.6 Tensile test

Tensile test was carried out on universal testing machine (UTM). In this test tensile force is applied on the material from both ends until the material breaks. This test is done to find ultimate tensile strength (UTS) and breaking load of the material. UTS is determined by plotting load on Y axis and displacement on X axis. In this project work ASTM standards was used to cut SMP samples to standardized dimensions. Before carrying tensile test all the samples were bend to check flexibility and the samples which does not break was used to find UTS this was done to avoid premature failure of the sample.

III. RESULTS AND DISCUSSION

3.1 Shape memory effect (SME)

The percentage of SME was calculated using SME = θm/180º-θe and the SME% is tabulated. The Shape Memory Effect obtained was in between 80% to 96%.

Table 3.1: Shape Memory Effect of SMP

Sample No.	Diameter d(mm)	Thickness t(mm)	Θ_m	Θ_e	SME%
1	30	1	125	25	80.65
2	30	1	120	35	82.76
3	30	1	130	40	92.86
4	30	1	130	45	96.63
5	30	1	125	50	96.15

3.2 Recovery ratio (RN)

The percentage of recovery ratio was calculated from RN = θ0- θN/ θ0 formula and the RN% is tabulated in below table. The recovery ratio obtained was in between 80% to 94%.

Table 3.2: Recovery ratio

Sample No.	Diameter d(mm)	Thickness t(mm)	Θ_0	Θ_N	R_N
1	30	1	125	25	80.00
2	30	1	130	21	83.85
3	30	1	120	15	85.50
4	30	1	135	12	91.12
5	30	1	140	08	94.29

3.3 Glass transition temperature

The glass transition temperature was determined by DSC for five SMP samples. Table gives glass transition temperature of SMP samples and transition temperature is as shown.

Table 3.3: Glass transition temperature of SMP samples

Sample No.	Glass transition temperature in degree Celsius
1	43.83
2	28.62
3	43.44
4	45.35
5	45.84

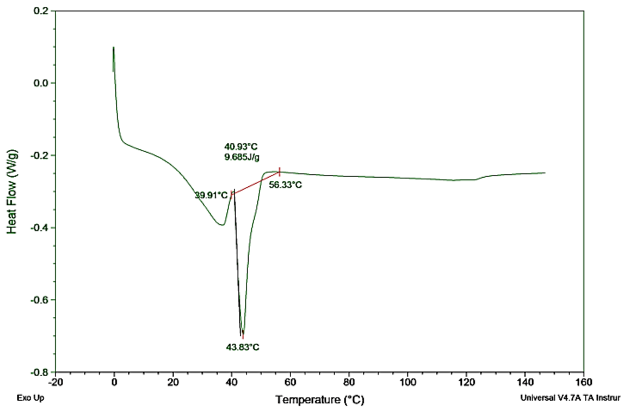

Fig. 3.1 (a): Glass transition temperature of sample 1

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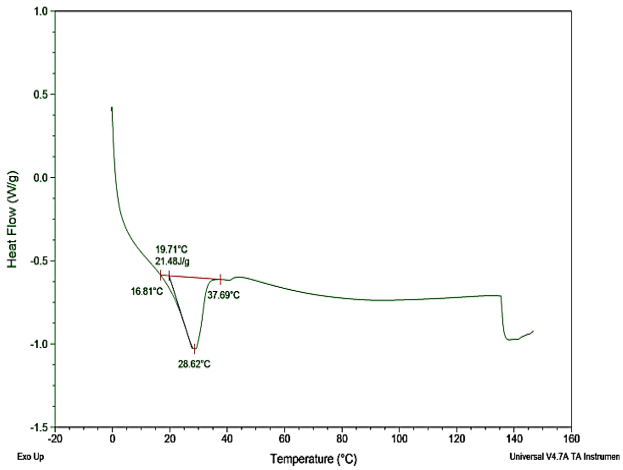


Fig. 3.1 (b): Transition temperature of sample 2

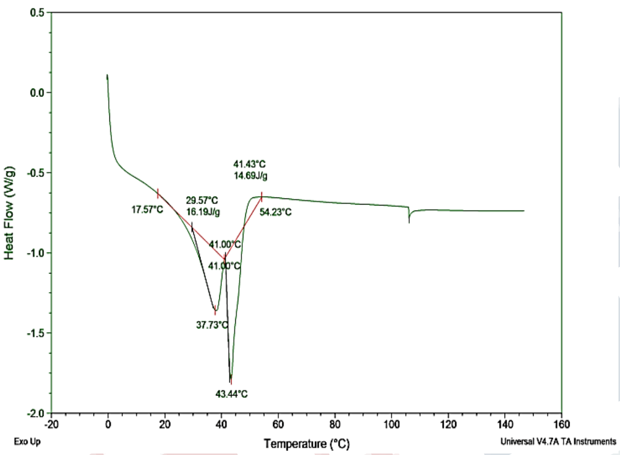


Fig. 3.1 (c): Transition temperature of sample 3

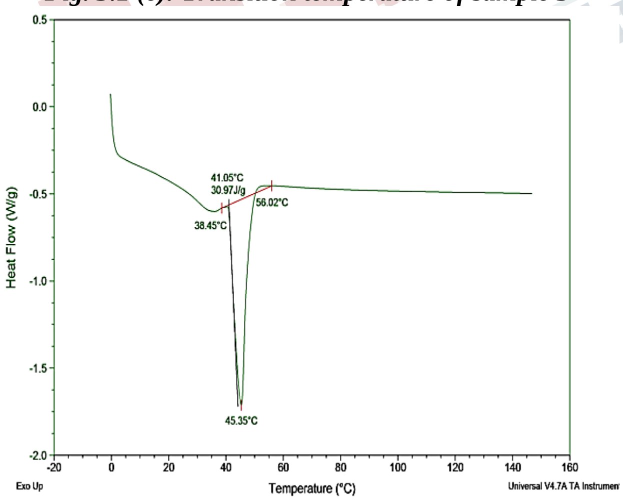


Fig. 3.1 (d): Transition temperature of sample 4

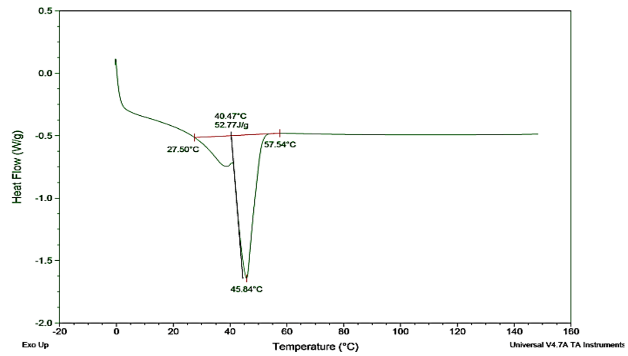


Fig. 3.1 (e): Transition temperature of sample 5

3.4 Tensile test

Tensile test was carried out to determine ultimate tensile strength (UTS) and breaking load. Table gives UTS and breaking load of all five SMP samples. The Load v/s Displacement curves under tensile load of samples are as shown in figs 3.2 (a) to 3.2 (d).

Table 3.4: UTS and Break load of all SMP samples

Sample No.	UTS in N/sq mm	Break load in N
1	5.181	13.00
2	0.994	30.598
3	3.455	32.167
4	5.815	30.304
5	1.923	30.990

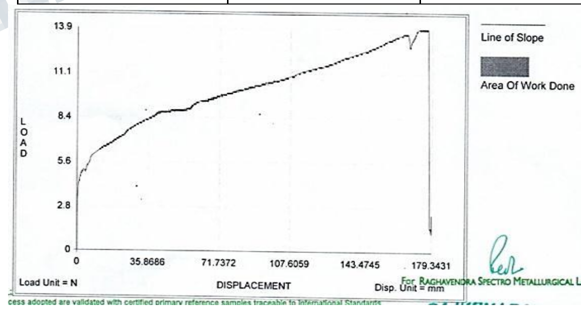


Fig. 3.2 (a): UTS and Break load of sample 1

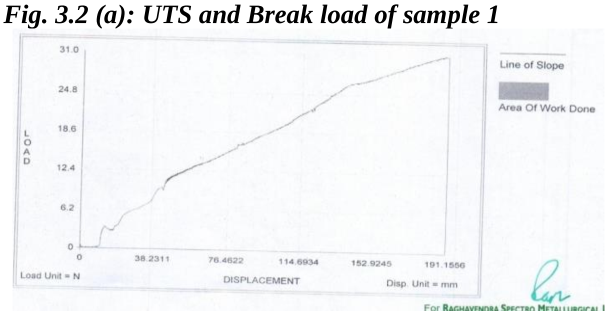
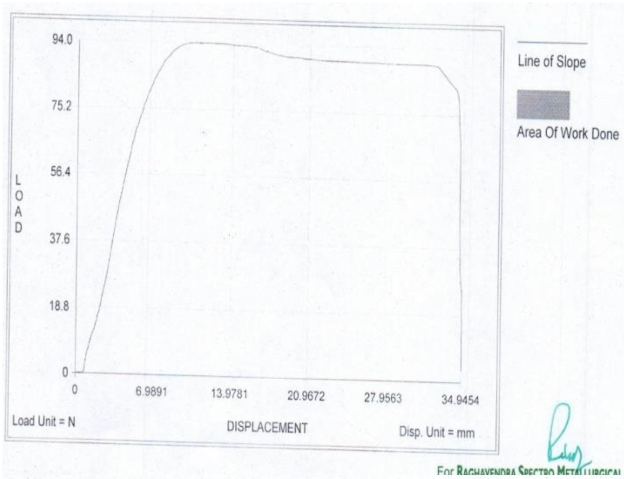


Fig. 3.2 (b): UTS and Break load of sample 2



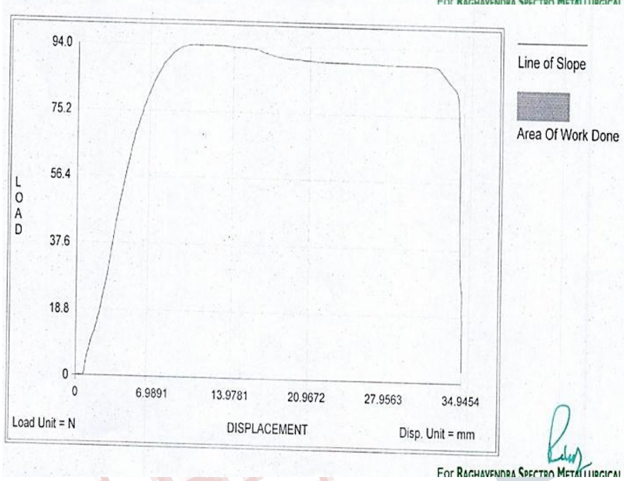


Fig. 3.2 (c): UTS and Break load of sample 3

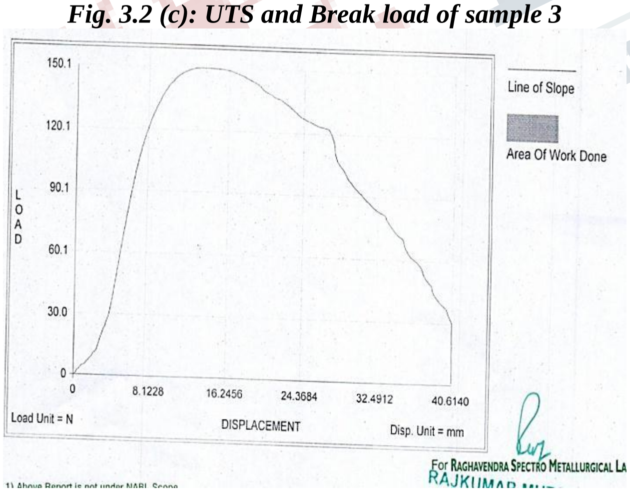


Fig. 3.2 (d): UTS and Break load of sample 4

It can be observed that the SMP exhibits good tensile strength and bear a tensile load of up to a maximum of 150N. The SMP also exhibits a good ductility as shown.

IV. CONCLUSIONS

1. The shape memory polymer was synthesized successfully.
2. The prepared SMP samples exhibits good shape memory effect between 80% to 96%.
3. The prepared SMP samples also exhibits good recovery ratio between 80% to 94%.
4. The SMP exhibits good glass transition temperature so that can be used in various applications conveniently.
5. The SMP exhibits good tensile strength as well as good ductility.

V. ACKNOWLEDGEMENT

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