3rd Polish Congress of Mechanics

21st International Conference on Computer Methods in Mechanics

Editors

Michał Kleiber
Tadeusz Burczyński
Krzysztof Wilde

Jarosław Górski
Karol Winkelmann
Łukasz Smakosz

Shape memory polymer – shape fixity and recovery in cyclic loading

Maria Staszczak[1], Elżbieta Pieczyska[2], Michał Maj[3], Katarzyna Kowalczyk-Gajewska[4], Mariana Cristea[5], Hisaaki Tobushi[6], Shunichi Hayashi[7]

[1,2,3,4] *Institute of Fundamental Technological Research PAS*
Pawińskiego 5b, 02-106 Warsaw, Poland
e-mail: mstasz@ippt.pan.pl [1], epiecz@ippt.pan.pl [2], mimaj@ippt.pan.pl [3], kkowalcz@ippt.pan.pl [4],

[5] *"Petru Poni" Institute of Macromolecular Chemistry*
Aleea Grigore Ghica Voda 41A, 700487 Iasi, Romania
email: mcristea@icmpp.ro

[6] *Aichi Institute of Technology*
1247 Yachigusa, Yakusa-cho, Toyota City, Aichi Prefecture 470-0392, Japan
email: tobushi@aitech.ac.jp

[7] *SMP Technologies Inc.*
Ebisu First Place1-22-8, Ebisu, Shibuya-Ku, 150-0013 Tokyo, Japan
email: hayashi@smptechno.com

Abstract

The paper concerns investigation of polyurethane shape memory polymer (SMP) properties. Shape fixity and shape recovery, important parameters for the SMP applications, were quantitatively estimated in thermomechanical cyclic loading; three subsequent thermomechanical loading cycles were performed. It was observed that the shape fixity is proper and does not depend on the cycle number. The obtained mean values of shape fixity parameters are 97-98 %. Although the shape recovery is poor (=83 %) in the first cycle of the thermomechanical loading, it is excellent in the subsequent cycles (=99-100 %). The evaluated parameters confirm good shape memory properties of the SMP.

Keywords: shape memory polyurethane, shape fixity, shape recovery, thermomechanical loading, cyclic loading

1. Introduction

Shape memory polymers (SMP) represent a class of smart materials which has competitive advantages compared to shape memory alloys: low weight, low cost, easy manufacturability, good shape fixity and recovery properties [1].

Shape memory polymers are able to response to a particular external stimulus, such as heat, light, moisture, etc.; however, the thermo-responsive SMPs are the most common.

The functional properties of the thermally responsive polymers are related to the glass transition temperature T_g, in which the characteristics of the polymer behaviour are affected by molecular motion that varies depending on the temperature. If a SMP is deformed at temperature above T_g and cooled down to temperature below T_g by holding the deformed shape constant, the deformed shape is fixed. If the shape-fixed element is heated up again to temperature above T_g under no-load conditions, it recovers its original shape and properties [2]. The SMP shape memory behaviour was investigated in the paper.

2. Materials and specimens

The material used in the performed experiment was the polyurethane shape memory polymer, characterised by T_g approximately equal to 45°C and degree of crystallinity of ≈5%.

A dynamic mechanical analysis (DMA) was carried out in order to characterize the shape memory polyurethane properties. The DMA allowed to obtain important polymer parameters, like glass transition temperature (T_g), as well as storage modulus (E'), loss modulus (E'') and loss factor ($tan\ \delta$), depending on temperature [3]. The DMA experiment was conducted during bending deformation, with frequency of force oscillation 1 Hz and heating rate 2 °C/min.

The results obtained in DMA suggest that the PU-SMP material fulfills some preliminary demands to perform as a shape memory polymer: a high glass elastic modulus E_g' (1250 MPa), proper value of the rubber modulus E_r' (12.1 MPa) and a high ratio of E_g'/E_r' (103). The T_g determined as $tan\ \delta$ peak is equal to 45°C.

3. Experimental procedure

The SMP specimens have been subjected to thermomechanical cyclic loading program performed on a MTS 858 testing machine equipped with a thermal chamber.

The general description of the thermomechanical loading program is presented in Tab. 1, while Fig. 1 shows the stress-strain curves obtained during the test.

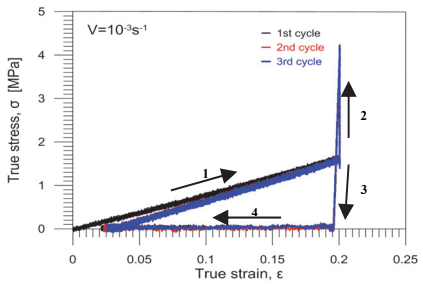

Figure 1: SMP true stress σ - true strain curves ε during thermomechanical test with strain rate $10^{-3}s^{-1}$. Number denotes process stage: 1 – loading at T_g+20°C, 2 – cooling down to T_g-20°C, 3 – unloading at T_g-20°C, 4 – heating to T_g+20°C

* The research has been carried out with the support of the Polish National Center of Science under Grant No. 2011/01/M/ST8/07754

At first, the specimen was heated to high temperature T_h = 65°C (T_g+20°C). Then, it was loaded at T_h till maximum strain of 20% with strain rate of $10^{-3}s^{-1}$ (Fig. 1 – 1st stage). While maintaining the strain, the specimen was cooled down to T_l=25°C (T_g-20°C); (Fig. 1 – 2nd stage). After that, the specimen was unloaded at T_l with the same strain rate (Fig. 1 – 3rd stage). During the subsequent heating from T_l to T_h under no-load conditions the SMP specimen almost recovered its original shape (Fig. 1 – 4th stage); however a residual strain ε_{ir} was recorded. The thermomechanical loading cycle was repeated three times.

Table 1: Description of thermomechanical loading program

Heating up to 65°C	Loading up to 20 % at T_h = 65°C	Cooling down to 25°C	Unloading to 0 N at T_l = 25°C	Heating up to 65°C
T	F	T	F	T

4. Results and discussion

Experimental results obtained during thermomechanical test carried out for SMP with strain rate of $10^{-3}s^{-1}$ are demonstrated in Fig. 2

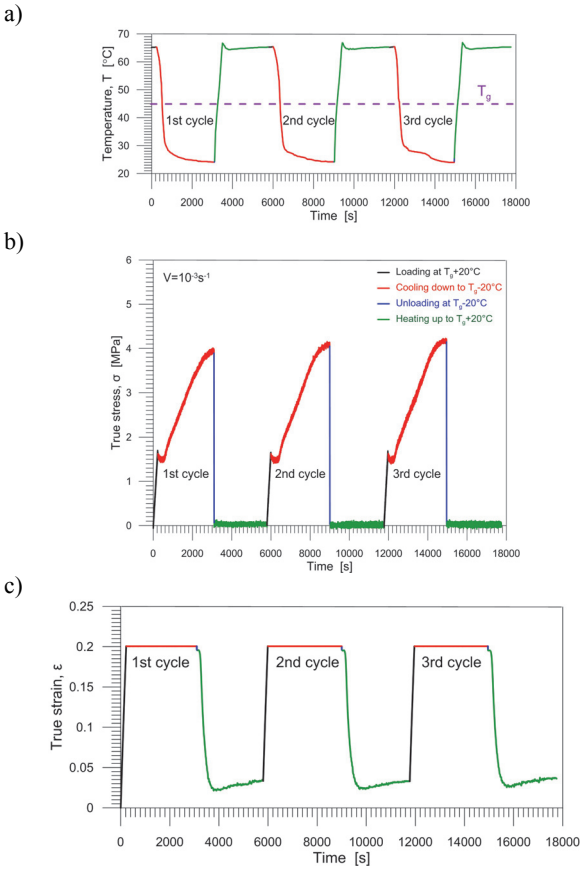

Figure 2: Experimental results obtained during three SMP thermomechanical loading cycles conducted with strain rate of $10^{-3}s^{-1}$: a) chamber temperature vs. time; b) true stress σ vs. time; c) true strain ε vs. time

Shape fixity shows the measure how a temporary deformed shape is fixed (stored), while shape recovery means the measure of how well the original permanent shape is recovered (or restored).

The following equations were used in order to define shape fixity R_f (1) and shape recovery R_r (2) parameters in the N-th cycle, respectively:

$$R_f = \frac{\varepsilon_{un}(N) - \varepsilon_{ir}(N-1)}{\varepsilon_{\max} - \varepsilon_{ir}(N-1)} \cdot 100\% \quad (1)$$

$$R_r = \frac{\varepsilon_{un}(N) - \varepsilon_{ir}(N)}{\varepsilon_{un}(N) - \varepsilon_{ir}(N-1)} \cdot 100\% \qquad R_f = \frac{\varepsilon_u}{\varepsilon_{max}} \cdot 100\% \quad (2)$$

where ε_{max} denotes maximum strain, ε_{un} – the strain obtained after unloading at T_l and ε_{ir} – irrecoverable strain, i.e. strain obtained after heating up to T_h under no-load conditions.

The parameters of shape recovery R_r and shape fixity R_f have been estimated for 3 subsequent thermomechanical cycles. An example of the obtained results is shown in Tab. 2. As can be seen in the table, the shape fixity has not changed markedly in subsequent cycles, while the shape recovery is poor for the first cycle and excellent for the subsequent cycles of the thermomechanical loading.

Table 2: Example of shape fixity and shape recovery parameters in three cycles obtained for SMP

Cycle No	Parameters, %	
	Shape fixity	Shape recovery
1	98	83
2	98	100
3	97	99

5. Conclusions

The cyclic thermomechanical analysis has been used to quantify the shape memory effects of the rigid polyurethane shape memory polymer (T_g = 45°C).

Important parameters, crucial for the SMP applications, have been evaluated in three thermomechanical loading cycles. The obtained average value of the shape fixity is 97-98 %, while the shape recovery is 83 % in first cycle and 99-100 % in subsequent thermomechanical loading cycles.

Estimation of the application parameters, i.e. shape fixity and shape recovery, carried out at maximum strain of 20 % and at temperature range T_g–20°C; T_g+20°C, gave reasonable values and confirmed good shape memory properties of the polymer.

References

[1] Tobushi, H., Matsui, R., Takeda, K., Pieczyska, E., *Mechanical Properties of Shape Memory Materials, Materials Science and Technologies*, NOVA Publishers, New York, 2013.

[2] Staszczak, M., Pieczyska, E.A., Maj, M., Kowalczyk-Gajewska, K., Cristea, M., Tobushi, H., Hayashi, S., Estimation of shape fixity and shape recovery – crucial parameters for shape memory polymer applications, *SolMech 2014: Proceedings of the 39th Solid Mechanics Conference,* Zakopane, Poland, pp. 267-268, 2014.

[3] Staszczak, M., Pieczyska, E.A., Maj, M., Kowalczyk-Gajewska, K., Cristea, M., Tobushi, H., Hayashi, S., Mechanical and infrared thermography analysis of shape memory polymer - focus on thermoelastic effect, *QIRT 2014: Proceedings of the 12th International Conference on Quantitative Infrared Thermography*, Bordeaux, France, pp. 183-1-9, 2014.