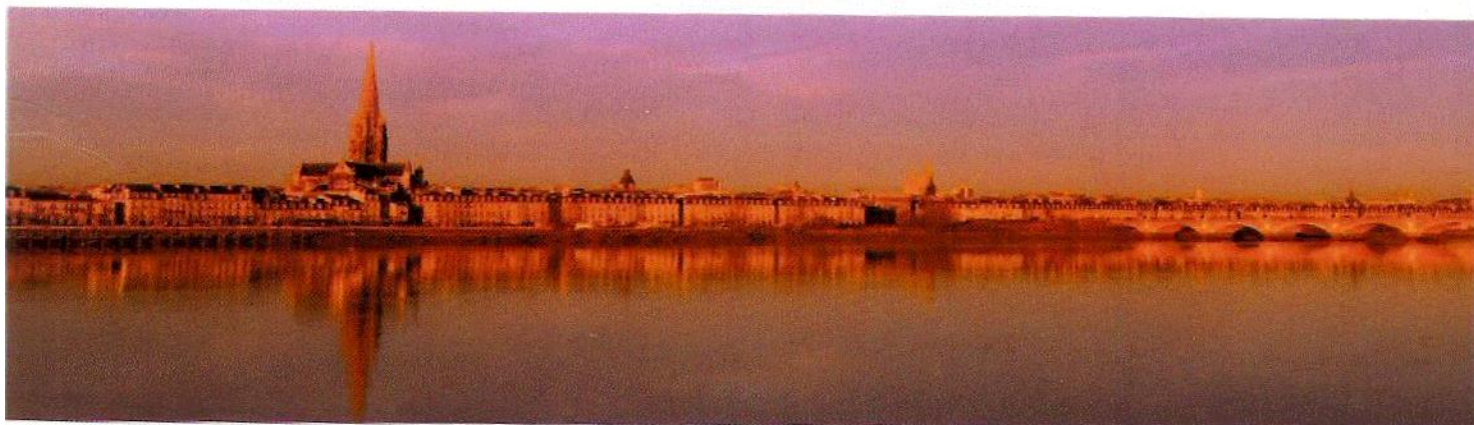


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Quantitative InfraRed Thermography

Mechanical and infrared thermography analysis of shape memory polymer - focus on thermoelastic effect

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Abstract

Experimental results of effects of thermomechanical couplings occurring in polyurethane shape memory polymer (PU-SMP) subjected to cyclic loading at different strain rates are presented. Stress-strain characteristics were recorded by the testing machine, whereas the specimen temperature changes were measured by a fast and sensitive infrared camera. The influence of strain rate on the polymer thermomechanical behaviour is studied. It was found that PU-SMP is very sensitive to the strain rate. The higher the strain rate, the higher the values of stress and temperature changes were obtained. In the initial stage of deformation a drop in temperature called thermoelastic effect was recorded determining a limit of the material reversible deformation.

1. Introduction

Dynamic development of technology, increasing demand for materials as well as awareness of the environment protection cause that recently a particular interest is observed on multifunctional smart materials, in particular on shape memory materials. In practice, it means that these materials are able to join the sensing and actuator functions which enable both innovative application and decrease of the device mass and sizes. The materials shape memory properties consist in recovery their original (permanent) shape from deformed (temporary) shape as a result of action of an external stimulus, such as temperature, magnetic/electric field, light with proper parameters, etc.

One of these materials is shape memory polymer (SMP). The mechanism of exhibiting shape memory property in polymer is definitely different than the one observed in shape memory alloys, because the crystallographic phase transition does not occur in polymers. Whereas, the functional characteristics of SMP, e.g. the rigidity, elastic modulus, coefficient of thermal expansion, change generally above and below its glass transition temperature T_g , since molecular motion of the polymer particles differs drastically below and above the T_g [1, 2].

Among various kinds of shape memory polymers the polyurethane (PU-SMP) can be distinguished. The PU-SMP is currently of great practical interest because of its good mechanical and shape memory properties and also low weight, good shape fixity and recovery, easy production technology, the transition temperature which can be set around the room and human body temperature, as well as low cost in comparison to Ti-based shape memory alloys. These properties allow to use PU-SMP in different fields, e.g. medical, protection of food, textile, housing and aviation industries [2, 3].

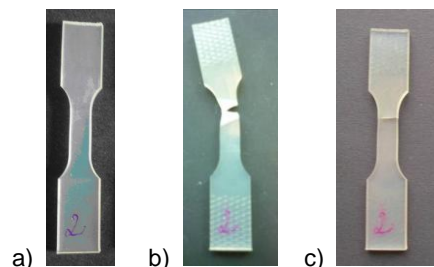


Fig. 1. SMP specimen at various stages manifesting shape memory properties a) before loading; b) after loading; c) after loading and subsequent heating at temperature $T_g + 20^\circ\text{C}$

The shape memory properties of PU-SMP are demonstrated in Fig. 1. The specimen taken off the grips of testing machine after deformation with high strain rate till the sample rupture is shown in Fig. 1b. When the shape-fixed specimen was heated under no load at temperature $T_g + 20^\circ\text{C}$ during 30 min, even so damaged specimen significantly recovered its original shape, however the decohesion is still noticed (Fig. 1a and 1c).

2. Material and specimens

The material used in the performed experiment was the polyurethane shape memory polymer, characterised by T_g approximately 45 °C and the degree of crystallinity of approximately 5 %. The parameters were derived from the differential scanning calorimetry (DSC). PU-SMP specimens of a gauge length of 15 mm were subjected to tension test. Technical drawing (a) and photograph (b) of the specimen are shown in Fig. 2.

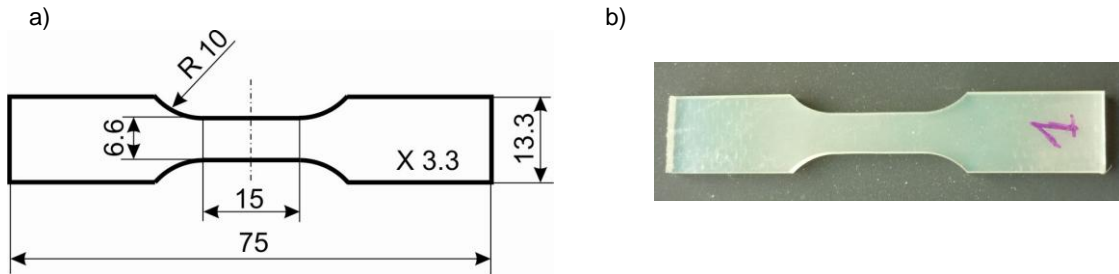


Fig. 2. Technical drawing (a) and photograph (b) of PU-SMP specimen for tension test

3. Dynamic mechanical analysis (DMA)

In order to get more information about this new shape memory polyurethane an additional thermo-mechanical test, called dynamic mechanical analysis (DMA), was carried out. 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 performed during bending deformation, with frequency of force oscillation 1 Hz and heating rate 2 °C/min. The obtained values of the modulus and loss factor vs. temperature are shown in Fig. 3. It can be noticed that the parameters are significantly changing depending on temperature; E' and E'' are high below T_g and low above the T_g . The value of $\tan \delta$ takes a peak at temperatures in the vicinity of T_g . Taking into account the modulus behaviour, four regions can be distinguished in this diagram: the glassy region (I), the glass transition region (II), the rubbery plateau region (III) and the flowing region (IV), respectively (Fig. 3).

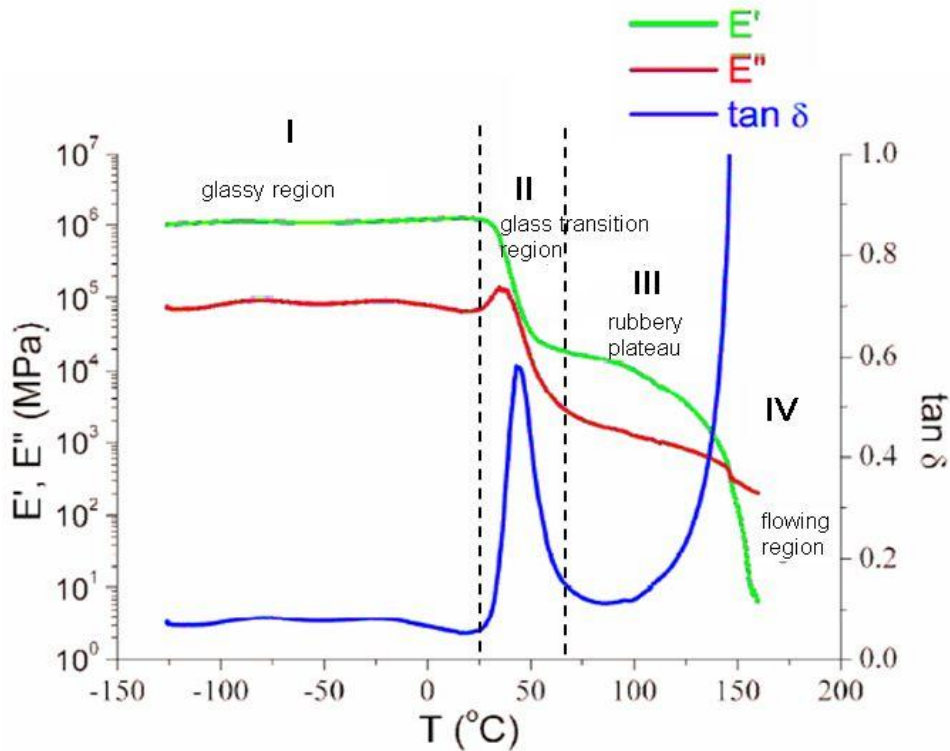


Fig. 3. DMA results obtained for the PU-SMP: variation of the storage modulus (E'), loss modulus (E'') and loss factor ($\tan \delta$) with temperature

Table 1. Results of DMA obtained for PU-SMP

Sample	E'_g (MPa)	T_g (determined as $\tan \delta$ peak) ($^{\circ}\text{C}$)	E'_r (MPa)	E'_g/E'_r
PU-SMP	1250	45	12.1	103

Results shown in Figure 3 and presented in Table 1 suggest that the PU-SMP material fulfills some preliminary demands to function as shape memory polymer. Namely, 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) were obtained. The T_g value determined as $\tan \delta$ peak is equal to approximately 45°C .

4. Experimental investigation of mechanical characteristics and temperature changes in PU-SMP in tension

SMP specimens (see Fig. 2) were subjected to tension test performed on MTS 858 testing machine at room temperature with various strain rates: 10^{-2}s^{-1} , 10^{-1}s^{-1} and 10^0s^{-1} . A fast and sensitive ThermoCam™ Phoenix infrared measurement system (IR) was used in order to measure in contactless manner the temperature changes accompanying the polymer deformation and to analyze experimentally effects of thermomechanical couplings [4-6]. The camera parameters are as follows: a wavelength range $3\ \mu\text{m} - 5\ \mu\text{m}$, maximal frequency used in this experiment 538 Hz, window size 160×256 pixels and thermal sensitivity equal to 0.02 K. The IR camera enables to find temperature distributions on the specimen surface, i.e. thermograms, which are useful in monitoring the nucleation and development of the localization process (Fig. 4). Moreover, the infrared measurement system allows determining the average temperature of the specimen during the deformation process which is usually presented in the diagrams (Figs 5-10) [7-8].

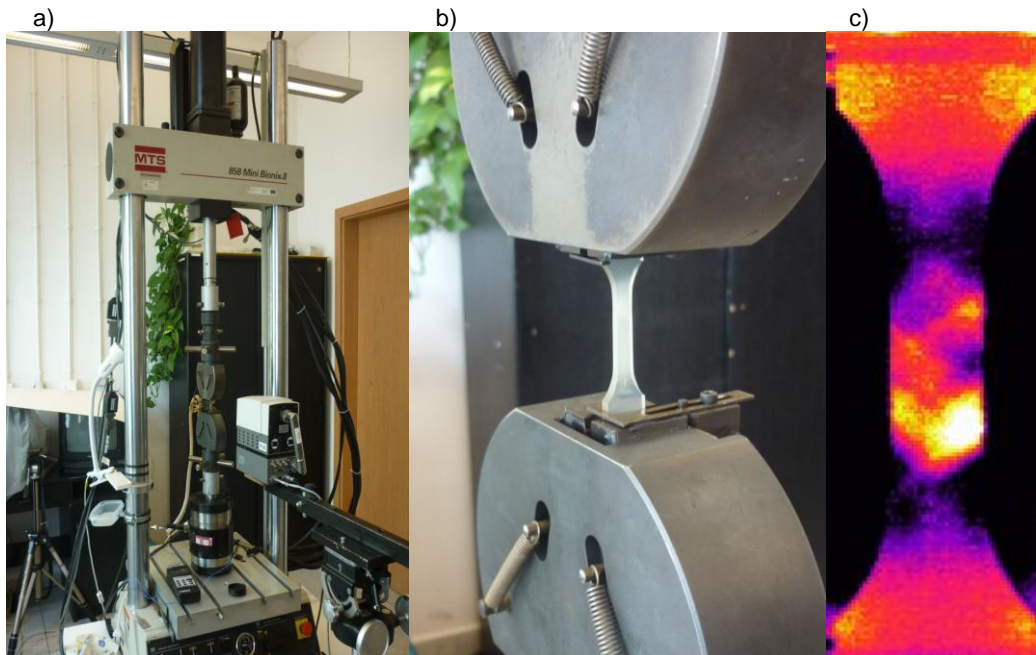


Fig. 4. a) Photograph of experimental set-up for investigation of mechanical and temperature characteristics of shape memory polymer, b) PU-SMP specimen in grips of testing machine, c) Infrared image of the specimen under deformation

5. Introduction to thermomechanical couplings

Thermomechanical couplings, i.e. the “strong” or “weak” interactions between the mechanical and temperature fields, play a significant role in nature, technology and our daily life, causing heating or cooling objects under loading and straining. The effects of thermomechanical couplings have been the subject of theoretical and experimental research, carried out by L. Kelvin [9], W. Nowacki, J.R. Klepaczko [10], A. Chrysochoos [11-12] and others, e.g. [7, 13].

Since the melting point and coefficient of thermal conduction are relatively low in polymers, the effects of thermomechanical couplings substantially influence their behaviour under different loadings. Strong thermomechanical couplings are observed in shape memory polymers subjected to loading, which is shown in Fig. 5. The stress and temperature vs. strain characteristics obtained during the PU-SMP tension enable to distinguish 3 different stages.

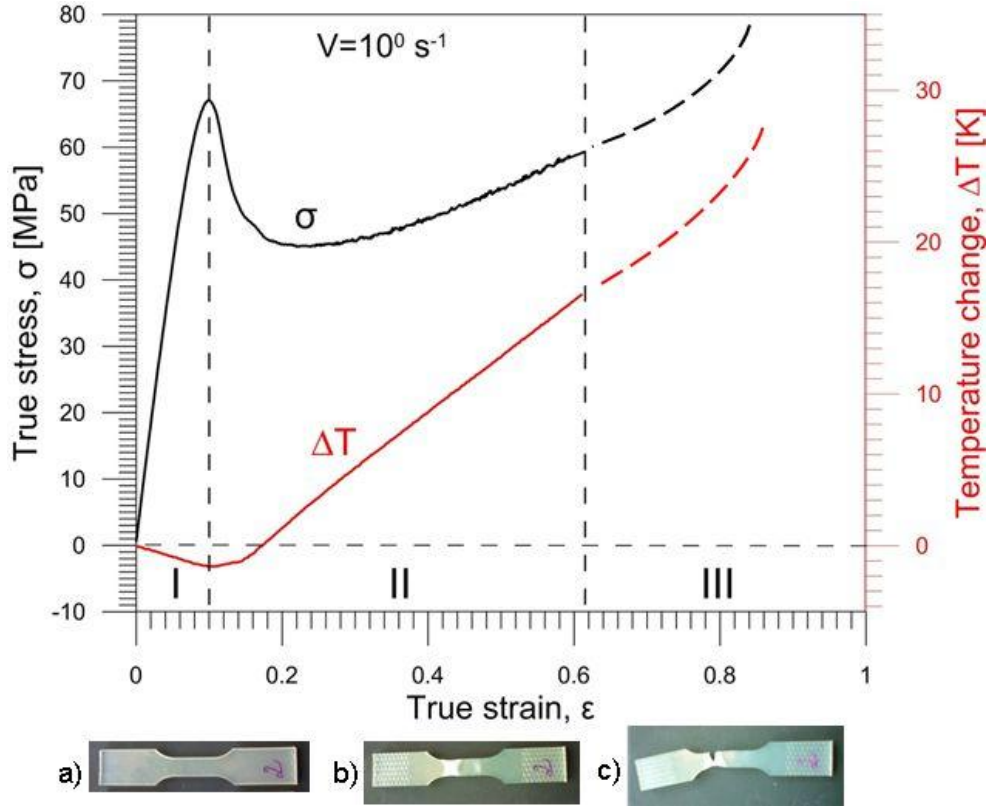


Fig. 5. Stress σ and temperature change ΔT vs. strain ϵ showing various stages of PU-SMP tension

The first (I) is the elastic stage of the reversible deformations, described by theory of elasticity. In the case of tension test a small drop in temperature is observed. The strain is low and the SMP specimen does not change significantly (Fig 5a).

The second (II) is the plastic stage, associated with change of the material structure, namely the polymer chains straightening. Moreover, the strong localisation effects can be observed (Fig. 5b). Like in metals, this deformation stage is characterised by a dissipative character. A significant increase of the sample temperature, depending on the strain rate, accompanies the deformation process. The polymers are very sensitive to the strain rate and their deformation is accompanied by significant temperature changes. The higher the strain rate, the more dynamic run of the deformation mechanisms and the larger temperature changes are observed.

Stage III is related to the mechanisms of damage (Fig. 5c). In this stage a breaking of the polymer chains occurs, leading to the specimen rupture. A huge increase of the specimen temperature is observed, especially in the rupture area. However, this stage, denoted by a dotted curve, is only an approximation of the stress and temperature changes.

6. Investigation of a limit of the polymer reversible material deformation - thermoelastic effect

Empirical identification of the boundary between the elastic and the plastic regimes of deformation is really complex. It is especially difficult to identify it in materials without pronounced yield point, like most of polymers. In this case, the methods based on the measurement of the specimen temperature under mechanical loading are usually very efficient. During tension, in the initial, elastic loading stage the temperature changes due to thermoelastic couplings are negative, while during the subsequent plastic deformation the temperature changes are always positive. The change of temperature ΔT of the specimen subjected to adiabatic uniaxial elastic deformation is called a thermoelastic effect and can be described by Kelvin equation as follows [9]:

$$\Delta T_{el} = - \frac{\alpha T \Delta \sigma_s}{c_p \rho}, \quad (1)$$

where α – the coefficient of linear thermal expansion, T – the sample absolute initial temperature, $\Delta \sigma_s$ – the isentropic change of stress, c_p – the specific heat at constant pressure, ρ – the material density. So, the value of maximal drops in the material temperature can be used for evaluating of a limit of the reversible material deformation.

An example of the stress (red line) and the related temperature change (black line) vs. strain obtained for the PU-SMP subjected to tension with strain rate 10^{-1}s^{-1} is shown in Fig. 6

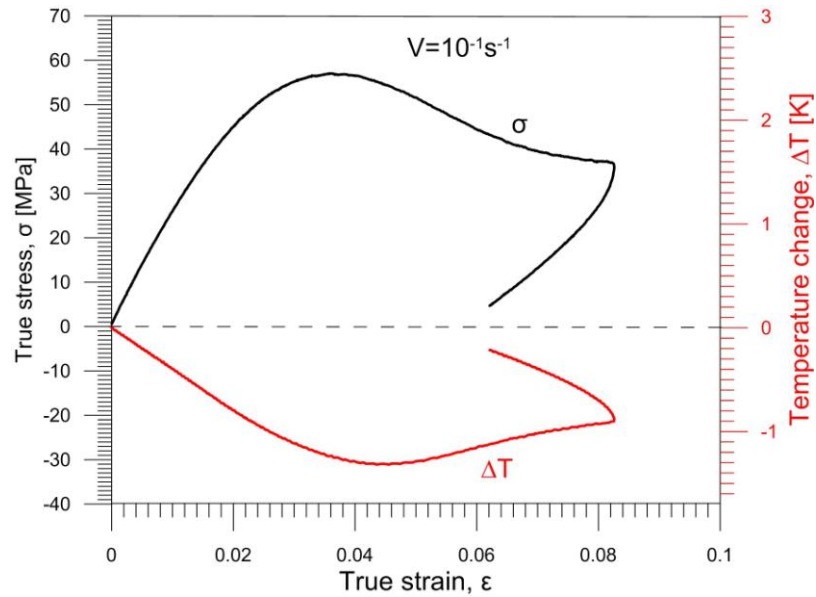


Fig. 6. Stress σ (black line) and the related temperature change ΔT (red line) vs. strain ϵ obtained for PU-SMP subjected to tension with a strain rate of 10^{-1}s^{-1}

The SMP was loaded until 8% of the true strain. After obtaining the maximal value (59 MPa) the stress decreases due to the structure changes and the localisation phenomena. The initial loading was accompanied by drop in temperature up to minus 1.3 K, followed by a smooth temperature increase caused by the dissipative mechanisms. During the unloading, the specimen temperature increases significantly.

Next figures present the stresses and their related temperature changes, obtained for the PU-SMP tension cyclic loading within strain approximately 6% carried out at room temperature with various strain rates.

The stress (black line) and the temperature change vs. strain (red line) for a strain rate of 10^{-2}s^{-1} are shown in Fig. 7a, while vs. time in Fig. 7b, respectively.

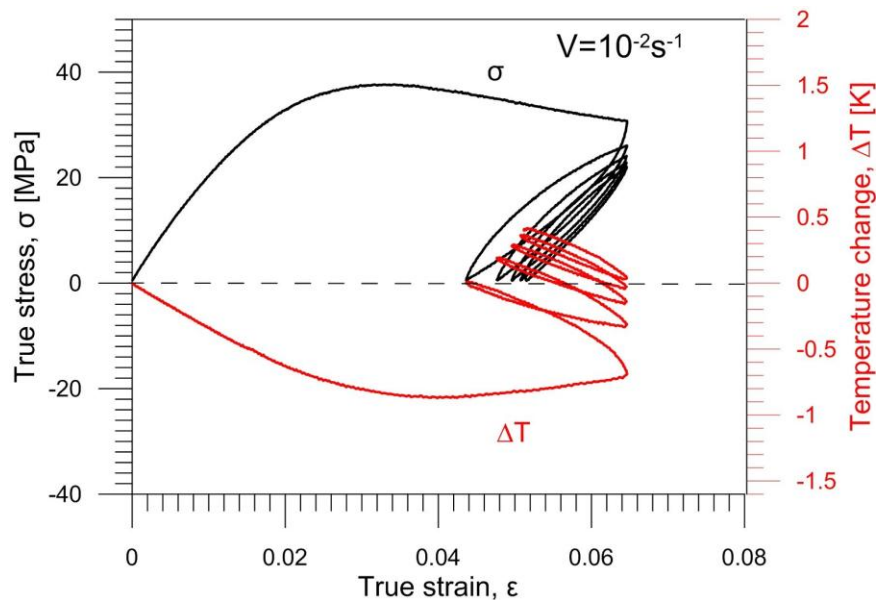


Fig. 7a. Stress σ (black line) and temperature change ΔT (red line) vs. strain ϵ for a strain rate of 10^{-2}s^{-1}

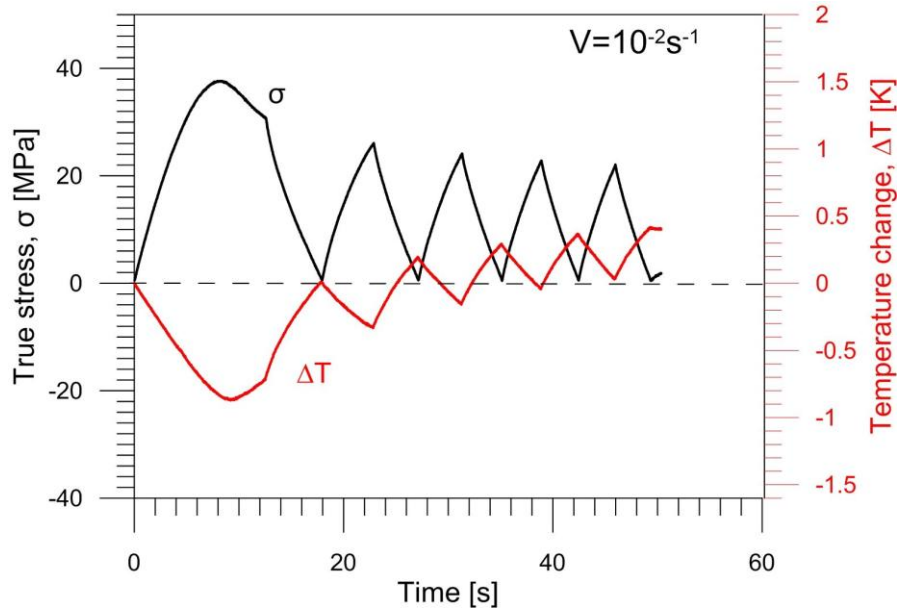


Fig. 7b. Stress σ (black line) and temperature change ΔT (red line) vs. time for a strain rate of $10^{-2} s^{-1}$

In order to estimate the influence of the strain rate, the next figures present results obtained for the 10 times higher strain rate, equal to $10^{-1} s^{-1}$ (Fig. 8).

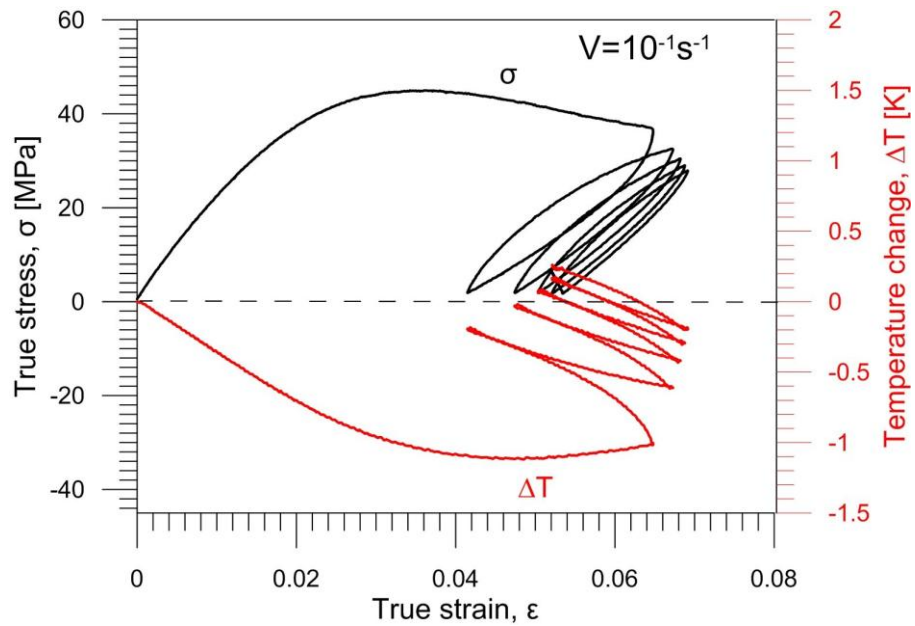


Fig. 8a. Stress σ (black line) and temperature change ΔT (red line) vs. strain ϵ for a strain rate of $10^{-1} s^{-1}$

The higher strain rate, the higher stress value and the higher temperature changes were obtained.

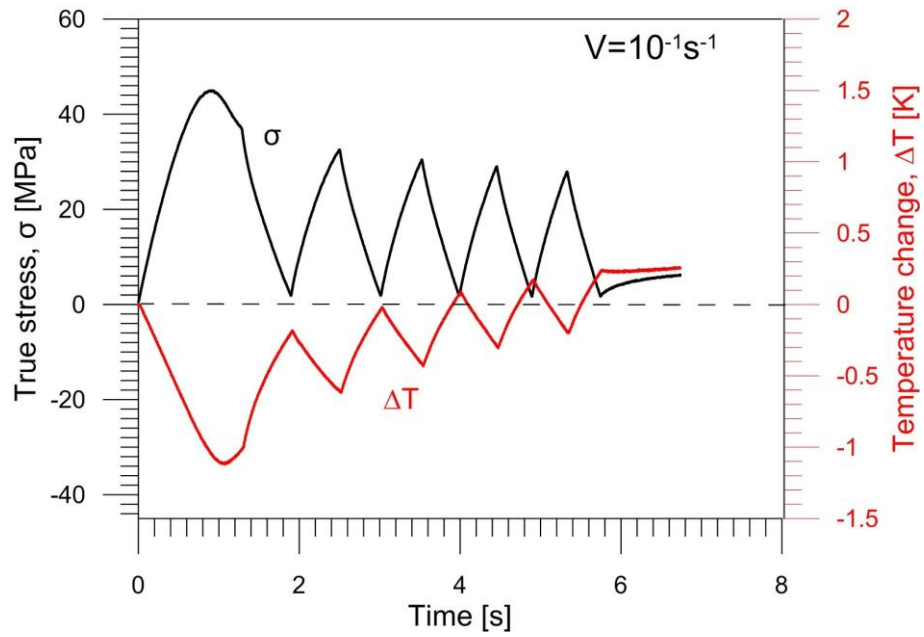


Fig. 8b. Stress σ (black line) and temperature change ΔT (red line) vs. time for a strain rate of 10^{-1}s^{-1}

Fig. 9 presents results obtained for much higher strain rate, equal to 10^0s^{-1} .

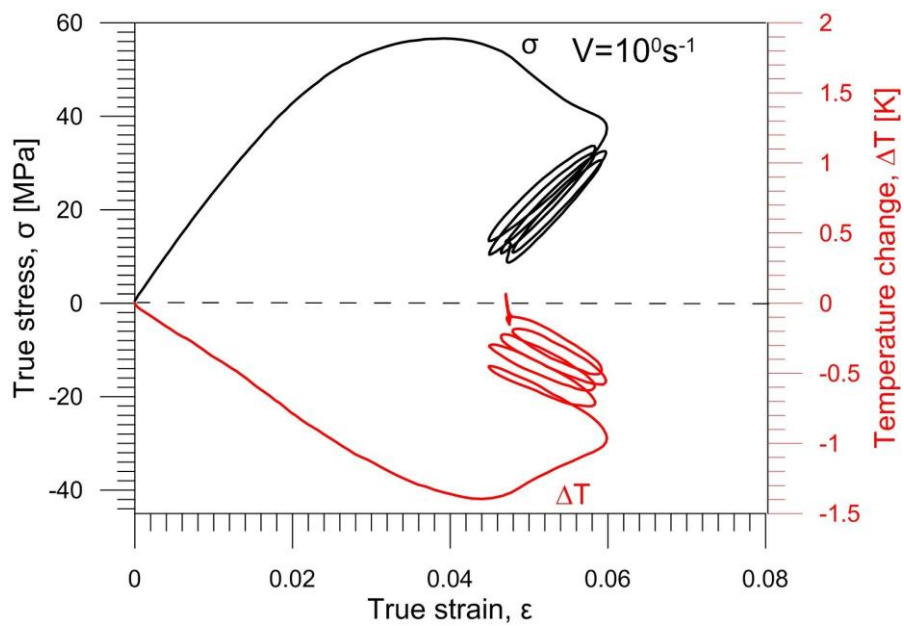


Fig. 9a. Stress σ (black line) and temperature change ΔT (red line) vs. strain ϵ for a strain rate of 10^0s^{-1}

For each strain cycle, a decrease in the specimen temperature during the initial (elastic) loading and the temperature increase during the unloading proces was observed. The obtained stress and the temperature changes manifest how huge is the influence of thermomechanical couplings on the PU-SMP mechanical behaviour.

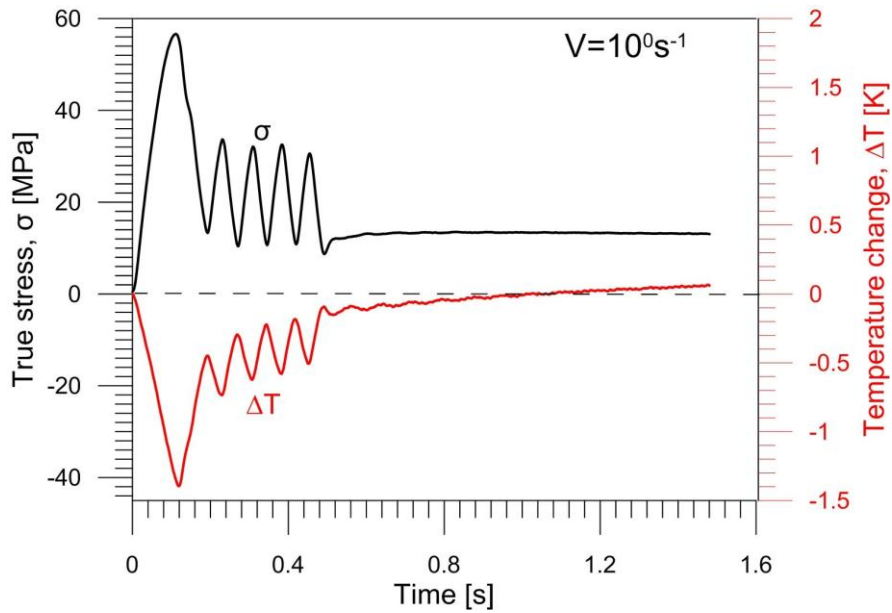


Fig. 9b. Stress σ (black line) and temperature change ΔT (red line) vs. time for a strain rate of 10^0 s^{-1}

Looking at the results obtained for various strain rates and presented in Figures 7-9 it can be noticed that at the higher strain rate not only the higher stresses and temperature changes were obtained, but also the difference between the first and the subsequent cycles of loading was significantly larger. This is caused by a great impact of the thermodynamic state of the PU-SMP specimen related to the first cycle of loading. At the higher strain rate the test conditions are close to adiabatic which significantly influence both the SMP structure and thermomechanical behaviour in the subsequent loading-unloading cycles. A comparison of the results presented in Figures 7 - 9, namely a stress and temperature changes vs. strain for cyclic loading carried out with various strain rates (10^{-2} s^{-1} , 10^{-1} s^{-1} and 10^0 s^{-1}) are shown in Fig. 10.

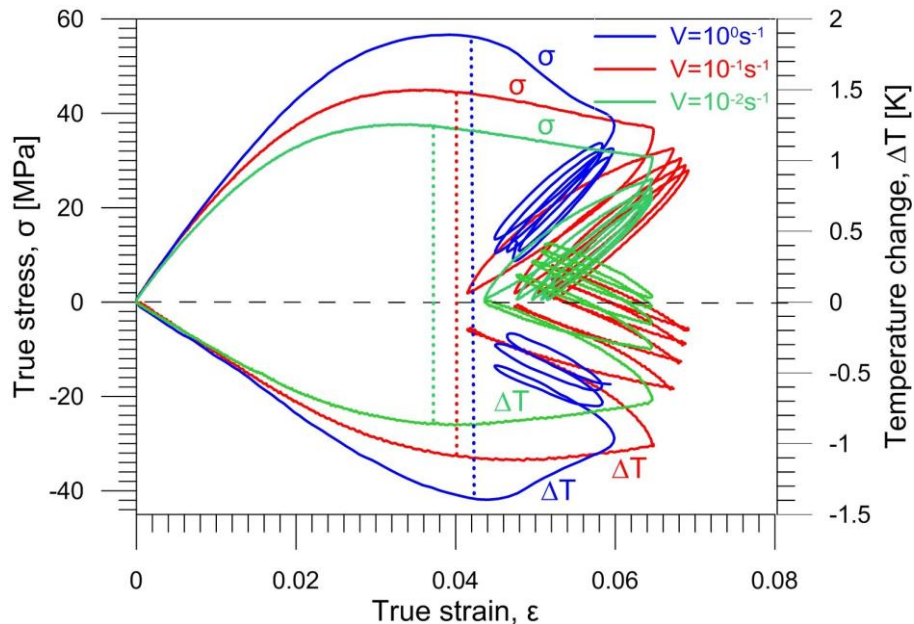


Fig. 10. Stress σ and temperature change ΔT vs. strain ϵ for SMP with strain rates: 10^{-2} s^{-1} , 10^{-1} s^{-1} and 10^0 s^{-1}

In the initial stage of strain (elastic) a decrease of the specimen temperature is observed. At higher strains the specimen temperature increases due to initiation and evolution of the dissipative mechanisms of plastic deformation. The change in the character of temperature evolution for the specimen during its loading allows determining a limit between the elastic and plastic regimes. It is observed that the higher strain rate the higher drops in temperature and the larger

yield points are obtained (Fig. 10). Using the values of maximal drops in temperature [13], limits of the PU-SMP reversible deformation were estimated as follows: 38 MPa for 10^{-2}s^{-1} , 45 MPa for 10^{-1}s^{-1} and 56 MPa for 10^0s^{-1} (Table 2).

Table 2. Yield point values estimated as maximal drops in temperature for PU-SMP at various strain rates

Strain rate	10^{-2}s^{-1}	10^{-1}s^{-1}	10^0s^{-1}
ΔT_{max} [K]	- 0.86	- 1.11	- 1.39
σ_{rev} [MPa]	38	45	56

7. Conclusions

Effects of thermomechanical couplings occurring during cycling tension loading of polyurethane shape memory polymer PU-SMP have been studied.

It was found that the PU-SMP is characterized by good mechanical and shape memory properties, as well as high sensitivity to the strain rate, manifested by both mechanical and thermal results.

The difference between the first and the subsequent cycles of loading was significantly larger. This is caused by a great influence of the PU-SMP thermodynamic state.

Values of limits of the reversible SMP deformation basing on its maximal drops in temperature within the initial stage of the loading were evaluated with a high accuracy. At higher strain rate higher values of the yield points were obtained.

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