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INFRARED THERMOGRAPHY ANALYSIS OF THERMOMECHANICAL SHAPE MEMORY POLYMER BEHAVIOR – INITIAL LOADING STAGE

M. Staszczak¹, E.A. Pieczyska¹, M. Maj³, K. Kowalczyk-Gajewska¹, D. Kukla¹, H. Tobushi² and S. Hayashi³

¹Institute of Fundamental Technological Research PAS, Warsaw, Poland, mstasz@ippt.pan.pl, epiecz@ippt.pan.pl; mimaj@ippt.pan.pl; kkowalcz@ippt.pan.pl

²AICHI Institute of Technology, Toyota, Japan, tobushi@aitech.ac.jp

³SMP Technologies Inc., Tokyo, Japan, hayashi@smptechno.com

Experimental results of effects of thermomechanical couplings occurring in polyurethane shape memory polymer (PU-SMP) subjected to cyclic loading at various 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 was studied. It was found that the SMP is very sensitive to the strain rate. The higher the strain rate, the higher the values of the stress and temperature changes were obtained. In the initial stage of deformation a drop in temperature called thermoelastic effect, determining a limit of the material reversible deformation, was investigated.

Introduction; material and specimens

Dynamic development of technology, increasing demand for materials as well as awareness of the environment protection cause that recently an increasing interest is observed in multifunctional smart materials, in particular in shape memory materials. The materials are able to combine the sensing and actuator functions which enable both innovative application and decrease of the device mass and sizes. 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, etc., change above and below its glass transition temperature T_g , since molecular motion of the polymer chains differs drastically below and above the T_g [1, 2]. Among various kinds of shape memory polymers the polyurethane (PU-SMP) can be distinguished due to its

good mechanical and shape memory properties, as well as low cost in comparison to Ti-based shape memory alloys. These properties allow using PU-SMP in different fields, e.g. biomedical, protection of food, textile, housing and aviation industries. The material used in the experiment was the polyurethane shape memory polymer, characterized by $T_g \approx 45^\circ\text{C}$ and the degree of crystallinity of $\approx 5\%$. Results obtained from dynamic mechanical analysis suggest that it fulfills preliminary demands to function as shape memory polymer. Namely, a high glass elastic modulus E_g' (1250 MPa), a proper value of rubber modulus E_r' (12.1 MPa) and a high ratio of E_g'/E_r' (103) were obtained.

PU-SMP subjected to loading - mechanical characteristics and temperature changes

PU-SMP specimens of a gauge length of 15 mm were subjected to tension tests 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 ThermaCamTM Phoenix infrared camera (IR) was employed in order to measure in

contactless manner the temperature changes accompanying the polymer deformation and to analyze effects of thermomechanical couplings [2-4]. The maximal frequency of the IR camera is 538 Hz, window size 160 x 256 pixels, a wavelength range 3-5 μm , thermal sensitivity 0.02 K. The camera enables obtaining the temperature distributions on the specimen surface, i.e. thermograms, which can monitor nucleation and development of the strain localization phenomena during the deformation (Fig. 1). Moreover, the IR system allows to determine a change of the SMP average temperature which is presented in the diagrams (Figs 1-5).

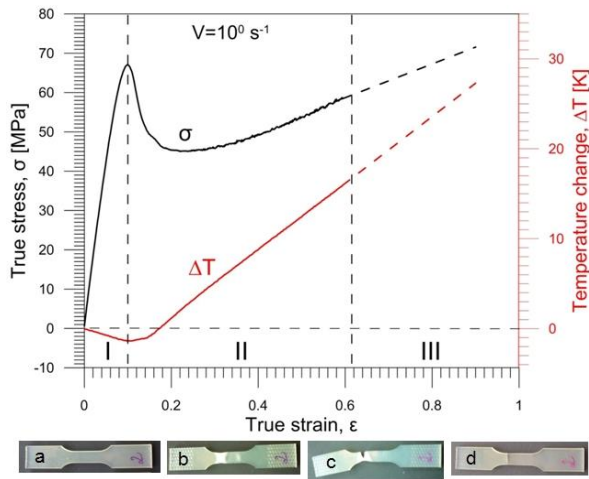


Fig.1. Stress σ and temperature change ΔT vs. strain ϵ and photos of various stages of the SMP tension.

The temperature changes of the SMP linked to the stress-strain curve enable distinguishing various stages of the SMP deformation process (Fig. 1). The first (I) is the reversible elastic stage of the deformations, described by theory of elasticity. In the case of tension, a small drop in temperature is observed, the strain is low and the SMP shape does not change significantly (Fig. 1a). The second (II) is the plastic stage, associated with change of the material structure, namely the polymer chains straightening. Moreover, strong localization effects can be observed (Fig. 1b). Like in metals, this deformation stage is characterized by a dissipative character [5], which in polymers can be related to strengthening of the chains. 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 are observed. The stage III is related to the damage mechanisms. In this stage a breaking of the polymer chains occurs, leading to the specimen rupture (Fig. 1c) and huge increase of its temperature, especially locally, in the rupture area [2]. The SMP specimen after loading, taking off the grips of testing machine and subsequent heating at $T_g+20^\circ\text{C}$, is shown in Fig. 1d.

Investigation of a limit of SMP reversible deformation - thermoelastic effect

Empirical identification of the boundary between the elastic and the plastic regimes of deformation is really complex, especially for 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. In the initial, elastic loading stage the temperature changes due to thermoelastic couplings are negative in tension, while positive during the subsequent plastic deformation. The temperature change of the specimen subjected to adiabatic uniaxial elastic deformation is called a thermoelastic effect and can be described by Kelvin formula (1):

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

where α denotes 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 a limit of the reversible material deformation. An example of the stress and the related temperature change vs. strain obtained for the PU-SMP subjected to tension with strain rate 10^{-1}s^{-1} (strain range 8%) is shown in Fig. 2.

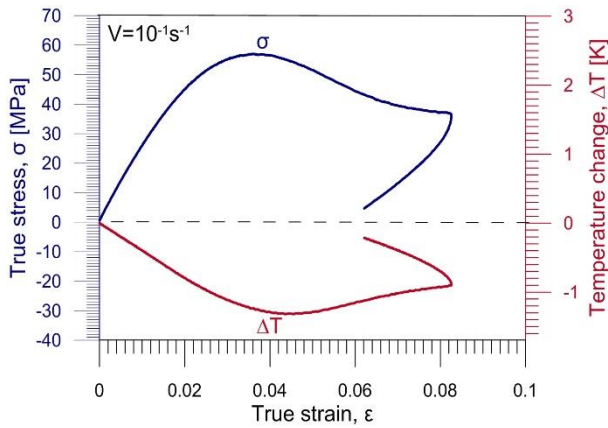


Fig. 2. Stress σ (blue line) and temperature change ΔT (red line) vs. strain ϵ for SMP tension with 10^{-1}s^{-1} .

After obtaining the maximal value (59 MPa) the stress decreases due to the structure changes and the localization phenomena. The initial loading was accompanied by a drop in temperature ≈ -1.3 K, followed by a smooth temperature increase caused by the dissipative

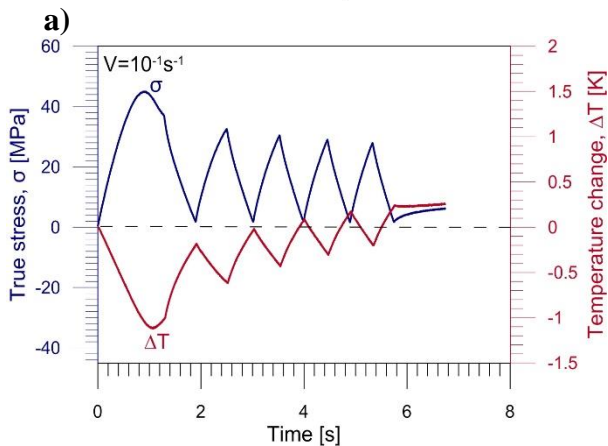
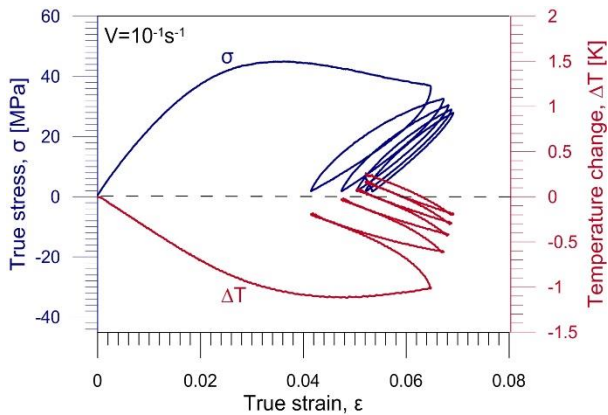


Fig. 3. Stress σ and temperature change ΔT for strain rate 10^{-1}s^{-1} : a) vs. strain ϵ ; b) vs. time.

mechanisms. During unloading, the specimen temperature increases significantly which is probably caused by superposition of the positive thermoelastic effect and exothermic effects of the SMP relaxation processes.

Next figures present the stresses and their related temperature changes, obtained for the PU-SMP tension cyclic loading within strain range approximately 6% carried out with various strain rates. The stress and the temperature change vs. strain for the strain rate 10^{-1}s^{-1} are shown in Fig. 3a, vs. time in Fig. 3b, respectively.

In order to estimate the influence of the strain rate, the test was also conducted for the 10 times higher strain rate 10^0s^{-1} (Fig. 4). The higher strain rate, the higher stress values and the higher temperature changes were obtained. 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.

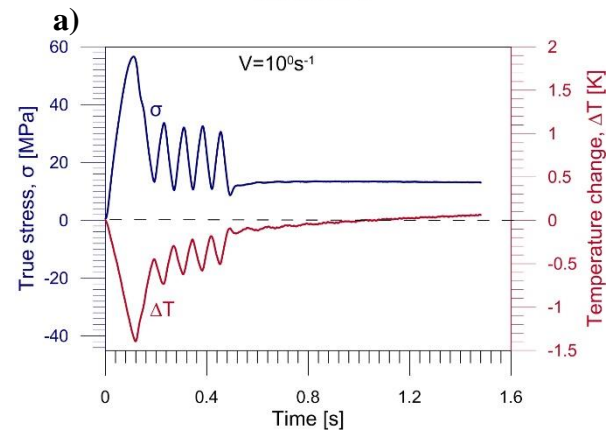
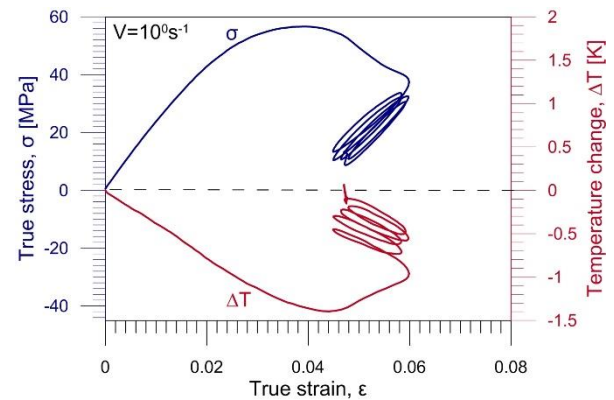


Fig. 4. Stress σ and temperature change ΔT for strain rate 10^0s^{-1} : a) vs. strain ϵ ; b) vs. time.

Looking at the results obtained for various strain rates presented in Figs 3-4 it can be noticed that at the higher strain rate not only the higher stresses and temperature changes have been obtained, but also the difference between the first and the subsequent cycles of the loading are significantly larger. This is caused by a huge impact of the initial thermodynamic state of the SMP specimen. At the higher strain rate the test conditions are closer to adiabatic which significantly influence both the SMP structure as well as its thermomechanical behaviour in subsequent loading-unloading cycles. A comparison of the results presented above, namely the stress and temperature changes vs. strain for cyclic loading carried out with various strain rates 10^{-2}s^{-1} , 10^{-1}s^{-1} and 10^0s^{-1} are shown in Fig. 5.

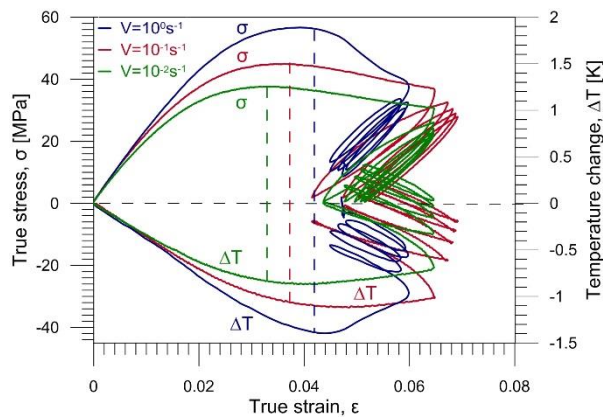


Fig. 5. Stress σ and temperature change ΔT vs. strain ϵ for SMP tension with strain rates: 10^{-2}s^{-1} , 10^{-1}s^{-1} and 10^0s^{-1} .

In the initial stage of strain a decrease of the specimen temperature is observed, while at higher strain the temperature increases due to initiation and evolution of the dissipative mechanisms of the deformation. Basing on the change in the character of the specimen temperature during its loading a limit between the elastic and the plastic regimes was determined. It was observed that at the higher strain rates higher temperature drops and larger yield points were obtained (Fig. 5). Using the values of maximal drops in temperature, limits of the reversible deformation were estimated (Table 1).

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

Table 1. Yield points estimated for PU-SMP

Conclusions

Effects of thermomechanical couplings during cycling tension in new multifunctional material PU-SMP were studied. It was found that the 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. Limits of the SMP reversible deformation were evaluated with a high accuracy due to advanced infrared technique. At the higher strain rate higher values of the yield points were obtained. The significant difference between the first and the subsequent loading cycles was caused by a great influence of thermodynamic state the initial SMP. The experimental results will be compared with the predictions obtained using the recently developed model [2].

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