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## Research paper / Praca doświadczalna

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# An analysis of the mechanical properties of HTPB-propellants using DMA

## Analiza właściwości mechanicznych paliwa na bazie HTPB z wykorzystaniem metody DMA

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**Abstract:** The paper presents the thermomechanical properties of solid rocket propellants containing hydroxyl-terminated polybutadiene. Dynamic mechanical analysis (DMA) was used in analysing the mechanical properties of propellant for two different sample geometries (cuboid and cylindrical). Non-isothermal and isothermal analyses were carried out in two holders: dual-cantilever and compression. The glass transition temperature of soft and hard segments in the propellants, the effect of dynamic force on sample strain, the creep-relaxation process (based on which parameters in the Burgers model were calculated) were determined based on the results of the analysis.

**Streszczenie:** W pracy przedstawiono właściwości termomechaniczne stałego paliwa raketowego zawierającego polibutadien zakończony grupami hydroksylowymi. Do analizy właściwości mechanicznych wykorzystano dynamiczną analizę mechaniczną (DMA) dla dwóch różnych geometrii próbek (prostokątnej i walcowej). Przeprowadzono badania nieizotermiczne i izotermiczne w dwóch uchwytach: podwójny wspornik i ściskający. Na podstawie przeprowadzonych badań określono: – temperaturę zeszklenia miękkich i twardych segmentów w paliwie, – wpływ siły dynamicznej na odkształcenie próbki, – proces pełzania-relaksacji (na podstawie którego obliczono parametry w modelu Burgersa).

**Keywords:** dynamic mechanical analysis, solid rocket propellant, glass transition temperature, compressive strength

**Słowa kluczowe:** dynamiczna analiza mechaniczna, stałe paliwo raketowe, temperatura zeszklenia, wytrzymałość na ściskanie

### Symbols and abbreviations

AP	ammonium perchlorate
Al	aluminium
DDI	dimeryl diisocyanate

DMA	dynamic mechanical analysis
DOA	dioctyl adipate
$\Delta E_a$	apparent activation energy [kJ/mol]
$E_1$	Young's modulus in Maxwell model [MPa]
$E_2$	Young's modulus in Kelvin-Voigt model [MPa]
$E'$	storage modulus [MPa]
$E''$	loss modulus [MPa]
$F_{stat}$	static force [N]
$f$	frequency [Hz]
$f_0$	pre-exponential frequency factor [Hz]
HTPB	hydroxyl-terminated polybutadiene
$L_0$	specimen length at ambient temperature [mm]
n-Fe <sub>2</sub> O <sub>3</sub>	nano-iron(III) oxide
$R$	gas constant [J/mol·K]
SN	sodium nitrate, NaNO <sub>3</sub>
$t$	time [s]
$T$	temperature [°C]
$T_1$	upper limit temperature [°C]
$T_g^{hard}$	glass transition temperature for hard segments [°C]
$T_g^{soft}$	glass transition temperature for soft segments [°C]
$T_{max}$	maximum temperature based on $E''=f(T)$ curves [°C]
$T_{onset}$	initial temperature based on $E'=f(T)$ curves [°C]
$T_{ref}$	lower limit temperature (reference temperature) [°C]
$\tan\delta$	mechanical loss modulus
$(\Delta L/L_0)$	change in specimen length in relation to its initial length [%]
$\varepsilon$	strain [ $\mu\text{m}$ ]
$\eta_1$	dynamic viscosity in Maxwell model [MPa·s]
$\eta_2$	dynamic viscosity in Kelvin-Voigt model [MPa·s]
$\sigma$	stress [MPa]
$\sigma_0$	applied stress [MPa]

## 1. Introduction

Heterogenous rocket propellants are formed from a multi-component suspension including an oxidizer, a metallic powder and a binding agent (a mixture of polymer, plasticizer and cross-linking agent). The most common materials used in heterogenous rocket propellants include ammonium perchlorate (AP), hydroxyl-terminated polybutadiene (HTPB), aluminium (Al), dibutyl phthalate, dioctyl adipate and different diisocyanates. The typical composition of heterogenous rocket propellants can be modified with different compounds to improve thermal, ballistic or performance properties. The parameters can be modified by using: burning rate modifiers, energetic polymers, energetic plasticizers or high-energetic materials [1-5].

Mechanical and viscoelastic properties of solid rocket propellants can be evaluated using a dynamic mechanical analysis (DMA) technique. Mechanical properties of propellants depend on the properties of polymer used in the binding agent, type and quantity of the cross-linking agent, and type and quantity of the filler (solids), phase transitions, relaxation or the solid's morphology [6] which is a consequence of chemical reactions and physical processes that take place over time, has significant effect on their relevant properties (*e.g.* chemical composition, mechanical properties, ballistic properties, *etc.* DMA consists of applying an oscillating force to the specimen and recording its response. The specimen can be deformed, *e.g.* by tension, shear, compression or bending. DMA measurements yield the following parameters: storage modulus ( $E'$ ), loss modulus ( $E''$ ) or mechanical

loss modulus ( $\tan\delta$ ). The storage modulus is an elastic response of the tested material and determines its ability to store energy. The loss modulus is a material response due to its viscous properties and the ability of a material to release thermal energy. The mechanical loss coefficient is a ratio of  $E''$  to  $E'$  and is the ability of a material to dissipate the energy as a result of internal friction or molecular rearrangement [7, 8]. Other parameters which can be determined using DMA include: tensile strength, elongation, stress, Young's modulus or compressive strength [9-11]. Wani *et al.* [12] which are viscoelastic in nature, are subjected to time, temperature, and frequency effects during the analysis to determine their dynamic and transient properties. The choice of parameters during the experiments like temperature, frequency, strain (%) tested the effects of different parameters on the properties of HTPB/AP/Al-based propellant in a dual-cantilever holder.

The study aimed to analyse the mechanical properties of heterogenous propellant containing hydroxyl-terminated polybutadiene using DMA.

## 2. Experimental section

### 2.1. Preparing the test samples

The materials used to obtain solid rocket propellants were: hydroxyl-terminated polybutadiene – HTPB R45M (Island Pyrochemical Industries), ammonium perchlorate – AP (Island Pyrochemical Industries), aluminium dust – Al (Benda-Lutz), dioctyl adipate – DOA (Boryszew Erg S.A.), lecithin (Sigma Aldrich), dimeryl diisocyanate – DDI (Island Pyrochemical Industries), sodium nitrate – SN (VWR Chemicals), nano-iron oxide – n-Fe<sub>2</sub>O<sub>3</sub>, catocene (Neo Organics). The composition of the resulting propellant was:

- 13.52% binding agent (9.99% HTPB, 1.52% DOA, 1.91% DDI, 0.10% lecithin),
- 71.48% oxidizers (43.28% AP, 28.20% SN),
- 12.00% Al,
- 3.00% burning rate modifier (1.50% n-Fe<sub>2</sub>O<sub>3</sub>, 1.50% catocene).

The propellant suspension was prepared in a NETZSCH laboratory planetary mixer. After mixing, the suspension was poured into a cuboidal mould and cured at 70 °C for 7 days. After curing, the propellant was used to prepare DMA test specimens in two shapes: 10.0×2.0×50 mm cuboid and Ø14.5×5.5 mm cylinder.

### 2.2. Test methods

The thermomechanical properties of propellants were analysed using a DMA apparatus – a NETZSCH-DMA 242 E Artemis. The specimens were tested in two holders: dual-cantilever and compression. In the dual-cantilever type holder, the ends of the cuboidal specimen are fixed and the middle section is pressed against the moving system section generating the oscillating force. Three strain areas: expansion, shear and compression were observed during the vibrations. Figure 1 shows the specimen in a dual-cantilever holder and its strain areas. The cylindrical specimen in the compression holder is compressed between the lower fixed section and the moving top section transferring the force exerted on the specimen surface.

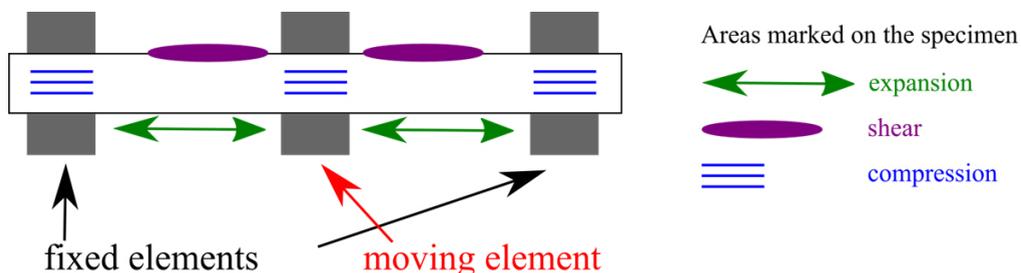


Figure 1. Specimen arrangement in the dual-cantilever holder

Measurements in the dual-cantilever holder were made under the following conditions:

- temperature range  $-120$  to  $100$  °C,
- temperature increase rate  $2$  °C/min,
- nitrogen flow rate  $50$  cm<sup>3</sup>/min,
- vibration frequency  $0.5$ ,  $1$  and  $2$  Hz,
- strain amplitude  $\pm 20$   $\mu\text{m}$  (DC1 procedure).

Measurements in the compression holder were made using three different procedures:

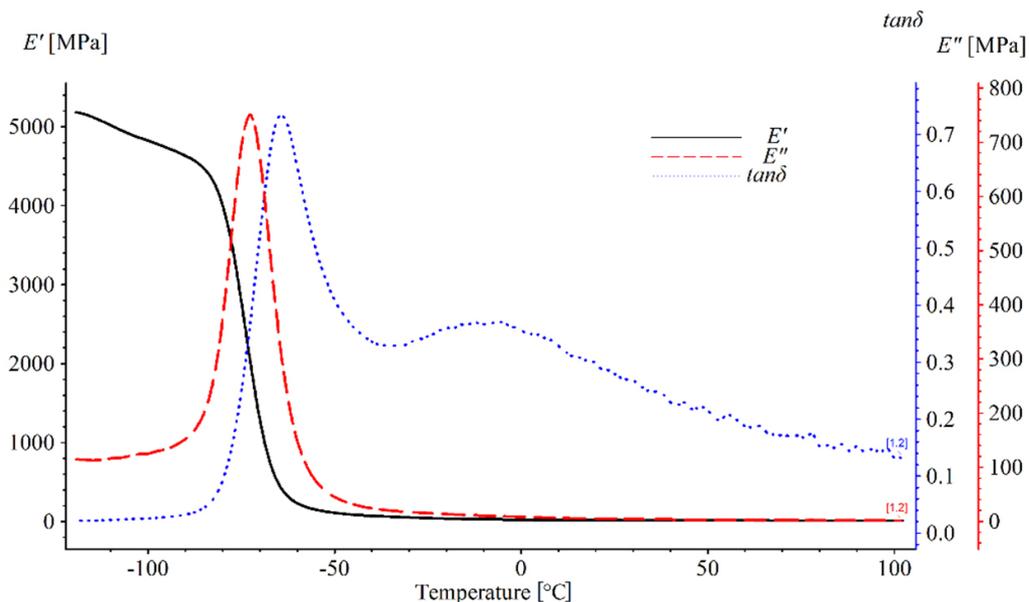
- The first procedure (C1) aimed to determine the coefficient of thermal expansion and was carried out under the following conditions:
  - temperature range  $-90$  to  $100$  °C,
  - temperature increase rate  $2$  °C/min,
  - nitrogen flow rate  $50$  cm<sup>3</sup>/min,
  - constant static force  $0.1$  N without dynamic force.
- The second procedure (C2) aimed to determine the effect of applied dynamic force on specimen strain. The tests were carried out under the following conditions:
  - constant temperature  $25$  °C,
  - vibration frequency  $1$  Hz,
  - dynamic force range  $0.1$  to  $9.2$  N with a force increase rate of  $0.01$ ,  $0.02$  and  $0.05$  N/s (three measurements were carried out at each rate).
- The third procedure (C3) aimed to test the creep-relaxation process and was carried out under the following conditions:
  - constant temperature  $25$  °C,
  - static force exerted for  $40$  min, followed by  $120$  min without the static force.

The following static forces were used in the measurements ( $F_{\text{stat}}$ ):  $25$ ,  $50$ ,  $100$ ,  $200$ ,  $500$ ,  $1000$ ,  $1500$  and  $4000$  mN.

### 3. Results and discussion

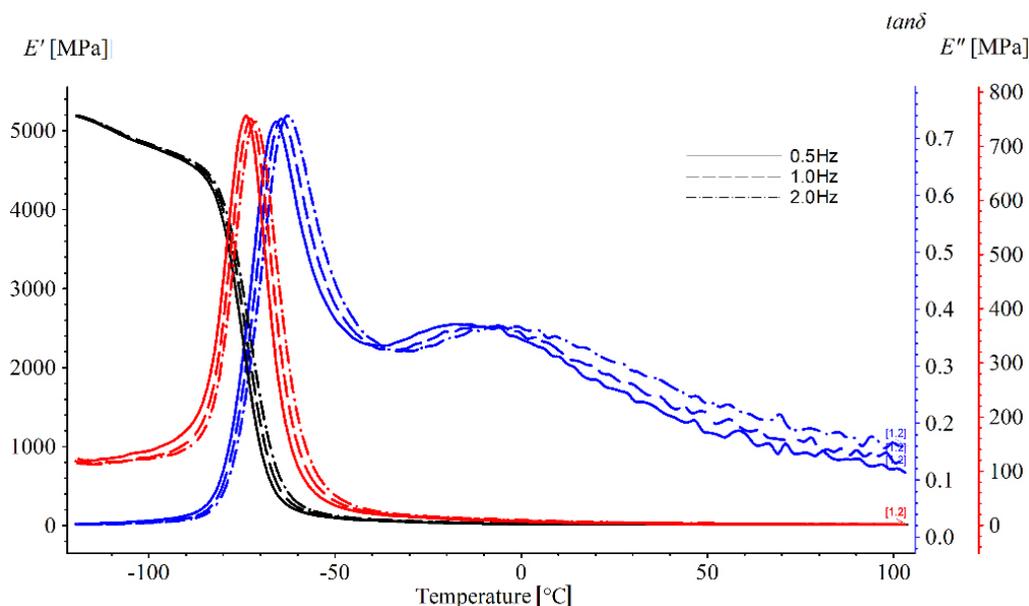
#### 3.1. Non-isothermal analysis – determination of glass transition temperature, storage modulus, loss modulus and mechanical loss modulus

The thermomechanical properties of the propellants can be characterized using the following DMA parameters: storage modulus, loss modulus and mechanical loss modulus. Figure 2 shows the relationship between the parameters and temperature (vibration frequency  $1$  Hz) for the tested propellant in accordance with procedure DC1.



**Figure 2.** Storage modulus ( $E'$ ), loss modulus ( $E''$ ) and mechanical loss modulus ( $\tan\delta$ ) as a function of temperature for the tested propellant (1 Hz)

The resulting DMA curves are typical of solid HTPB-based rocket propellants [12-15] which are viscoelastic in nature, are subjected to time, temperature, and frequency effects during the analysis to determine their dynamic and transient properties. The choice of parameters during the experiments like temperature, frequency, strain (%). Storage modulus decreases with temperature to 5185 MPa at  $-120\text{ }^{\circ}\text{C}$  and to 11 MPa at  $100\text{ }^{\circ}\text{C}$ . The storage modulus curve shows a single peak at  $-72.6\text{ }^{\circ}\text{C}$  ( $E'' = 750\text{ MPa}$ ). Two peaks can be observed in the third curve [ $\tan\delta = f(T)$ ]. The maximum temperature of the first peak is  $-64.4\text{ }^{\circ}\text{C}$ , and the second peak is  $-6.1\text{ }^{\circ}\text{C}$ . The second peak is wide and has a lower intensity than the first peak. This behaviour is related to the presence of hard and soft segments in polyurethane and the propellant. The first peak is a glass transition temperature of the soft segments ( $T_g^{\text{soft}}$ ) which determines the beginning or the end of the movement of the segments of the main HTPB chain. The second peak is related to a complex process involving two mechanisms: the interactions of the polymer itself and the interactions of the heterogenous material. It is a glass transition temperature of hard and/or soft segments, with the mobility significantly reduced due to solid content and interactions between the binding agent and the solids ( $T_g^{\text{hard}}$ ). The polyurethanes have a segmented structure – the hard segments being the urethane groups ( $-\text{N}=\text{C}=\text{O}$ ), and the polybutadiene chain being the soft segment [14-17].



**Figure 3.** The storage modulus ( $E'$ ), loss modulus ( $E''$ ) and damping ( $\tan\delta$ ) at different frequencies (0.5, 1.0 and 2.0 Hz)

**Table 1.** The determined DMA curve parameters for the tested rocket propellant ( $T_{\text{onset}}$  – initial temperature based on  $E' = f(T)$  curves, storage modulus at  $-100$  °C,  $T_{\text{max}}$  – maximum temperature based on  $E'' = f(T)$  curves, loss modulus at maximum temperature, glass transition temperature of soft and hard segments and mechanical loss modulus for soft and hard segments at their glass transition temperatures)

Parameter	Value for 0.5 Hz	Value for 1.0 Hz	Value for 2.0 Hz
$T_{\text{onset}}$ [°C]	-81.0	-80.2	-79.6
$E'$ at $-100$ °C [MPa]	4808	4825	4838
$T_{\text{max}}$ [°C]	-73.9	-72.6	-71.3
$E''$ at $T_{\text{max}}$ [MPa]	756	750	745
$T_{\text{g}}^{\text{soft}}$ [°C]	-65.8	-64.4	-62.8
$\tan\delta$ (soft segments) at $T_{\text{g}}^{\text{soft}}$	0.729	0.735	0.740
$T_{\text{g}}^{\text{hard}}$ [°C]	-17.6	-6.1	-1.1
$\tan\delta$ (hard segments) at $T_{\text{g}}^{\text{hard}}$	0.374	0.371	0.370

DC1 procedure was used to carry out measurements at different frequencies. Figure 3 shows the obtained DMA curves. An increase in the measurement frequency shifts all curves in the direction of higher temperatures. Based on the DMA curves for different vibration frequencies, the following parameters were used:

- $T_{\text{onset}}$  – initial temperature based on  $E' = f(T)$  curves,
- storage modulus at  $-100$  °C,
- $T_{\text{max}}$  – maximum temperature based on  $E'' = f(T)$  curves,
- loss modulus at maximum temperature,
- glass transition temperature of soft and hard segments and mechanical loss modulus for soft and hard segments at their glass transition temperatures.

Table 1 shows the parameters determined for each measurement. The relationship between glass transition temperature and vibration frequency can be expressed as:

$$f = f_0 \cdot \exp\left(\frac{aE_a}{R \cdot T}\right) \quad (1)$$

where:  $f$  – frequency,  $f_0$  – pre-exponential frequency factor,  $aE_a$  – apparent activation energy,  $R$  – gas constant,  $T$  – temperature.

Based on the glass transition temperatures of soft and hard segments, for each vibration frequencies using Equation 1, an apparent activation energy was determined. The apparent activation energy for soft segments is  $160 \pm 11$  kJ/mol, and for hard segments it is  $51 \pm 7$  kJ/mol. A similar relationship between the apparent activation energy for soft and hard segments can be observed for other HTPB-based propellants [14, 15, 18].

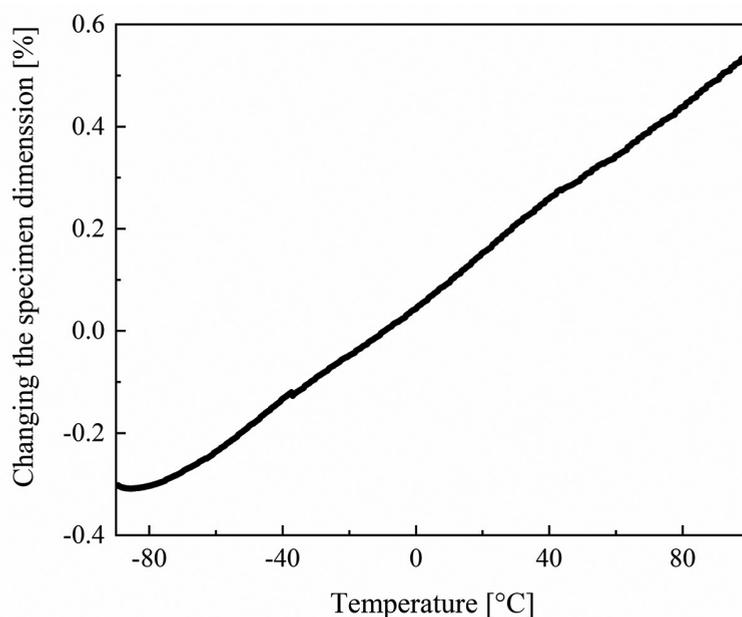
### 3.2. Non-isothermal analysis – determining the coefficient of thermal expansion

Based on the DMA measurements in the compressive holder, the coefficients of thermal expansions for the tested propellant (C1 procedure), were determined. The average coefficient of thermal expansion ( $\alpha$ ) within the range of temperatures from  $T_{ref}$  to  $T_1$  is defined as:

$$\alpha(T_1, T_{ref}) = \frac{\frac{\Delta L}{L_0}(T_1) - \frac{\Delta L}{L_0}(T_{ref})}{T_1 - T_{ref}} \quad (2)$$

where:  $L_0$  – specimen length at ambient temperature,  $T_1$  – upper limit temperature,  $T_{ref}$  – lower limit temperature (reference temperature),  $(\Delta L/L_0)$  – change in specimen length in relation to its initial length.

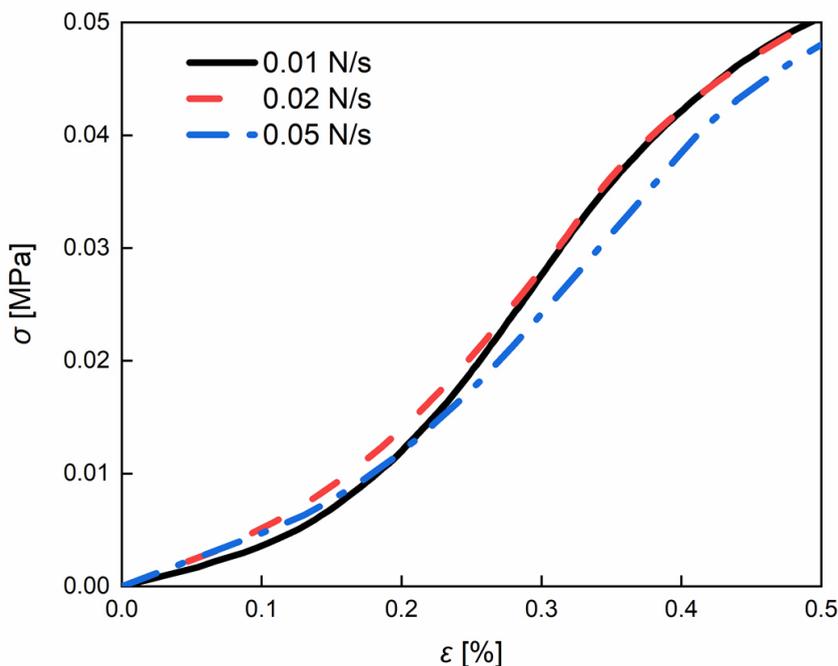
Figure 4 shows a typical specimen dimension vs. temperature curve. An increase in temperature increases the dimensions – a wide temperature range of  $-85$  to  $90$  °C displaying a linear change in specimen dimensions, being observed. The determined coefficient of thermal expansion within the temperature range  $-85.0$  to  $95.0$  °C is  $(51 \pm 4) \cdot 10^{-6}$  1/K.



**Figure 4.** The relationship between specimen dimensions and temperature for the tested propellant

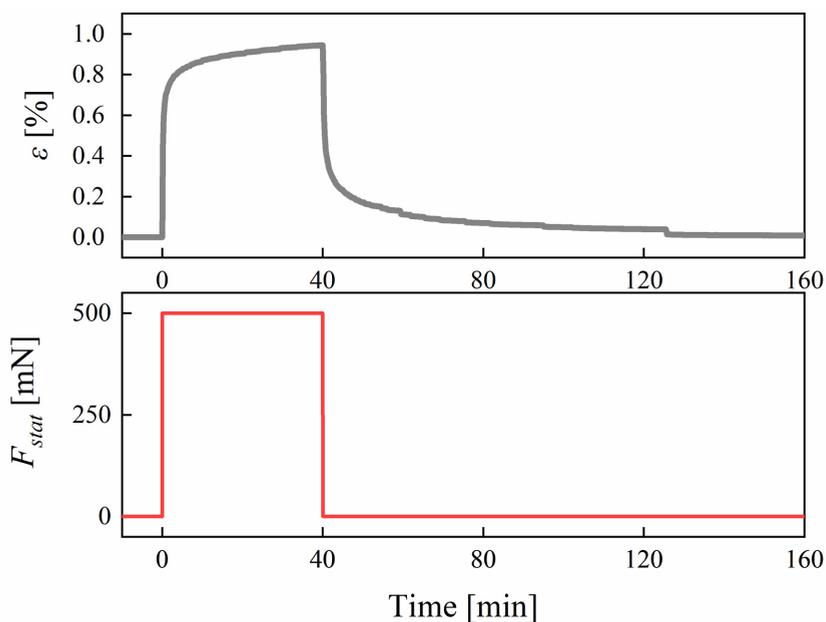
### 3.3. Isothermal analysis – determining the relationship between stress and strain and creep-relaxation curve

Based on the DMA measurement carried out using the C2 procedure using the compressive holder, a relationship between stress and strain of the tested specimen at different dynamic force increase rates, was determined (Fig. 5). A non-linear relationship between strain and stress within the tested stress range (0 to 0.05 MPa), was observed. A dynamic force increase rate does not significantly affect the strain from 0 to 0.015 MPa, and for higher values, the smallest specimen strain was observed for the highest dynamic force increase rate (0.05 N/s). The maximum specimen strain under the tested conditions was 0.5%.



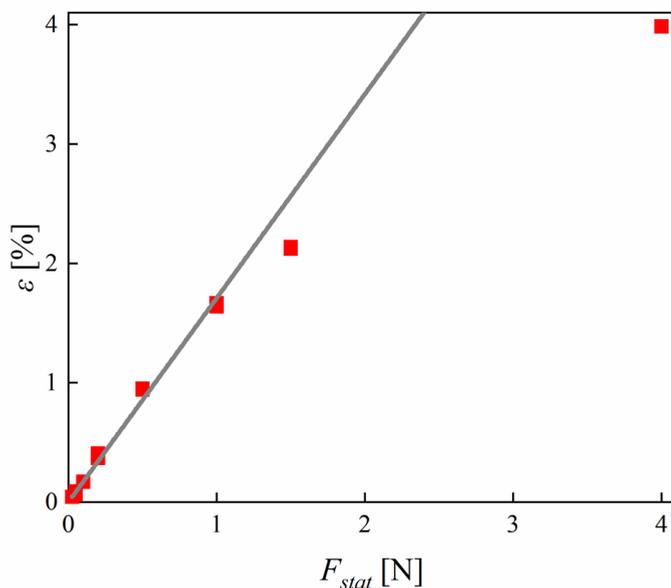
**Figure 5.** Stress ( $\sigma$ ) to strain ( $\epsilon$ ) relationship for different dynamic force increase rates

The creep and relaxation process was evaluated for the tested propellant at 25 °C, in accordance with C3 procedure. Figure 6 shows the creep-relaxation curve for a 500 mN static force (the first stage – 40 min). The long-term fixed stress results in a significant strain ( $\epsilon$ ) of the specimen (creep) at the beginning of the process – for the first 10 min, the specimen strain being 0.87%. The specimen strain was lower after that, the strain having increased by 0.07% for the next 30 min. For the following 120 min, the strain was recorded without static force – a relaxation process. For the first 20 min without static force, the strain was reduced by 0.83%, and after 120 min, the specimen practically returned to its initial dimensions.



**Figure 6.** The creep-relaxation curves for the tested specimen

To determine a linear area of viscoelastic properties of the tested propellant, a series of creep-relaxation measurements at different static forces (from 25 to 4000 mN), was carried out. The results were used to determine the strain ( $\varepsilon$ ) after 40 min of static force and based on these results, the relationship of the parameters and the static force were determined and are shown in Figure 7.



**Figure 7.** The relationship between strain ( $\varepsilon$ ) and static force ( $F_{stat}$ ) in the creep process

A linear range was obtained between 25 and 1000 mN, and for higher static force values, the strain is lower than the determined linear regression. The experimental points within 25-1000 mN were defined using the following linear equation ( $R^2 = 0.996$ ):

$$\varepsilon = 1.71 \cdot F_{\text{stat}} \quad (3)$$

The viscoelastic properties of materials are usually defined using different mechanical models. The basic model elements include: a perfect elastic body (shown as a spring), which follows Hooke's law and a perfect viscous body (shown as a dashpot) which follows Newton's law. The diagram, including a random combination of two elements, is used to model the behaviour. The two simplest models, Maxwell and Kelvin-Voigt, include two elements (spring and dashpot) and are referred to as two-parameter models. The Maxwell model is used to describe the relaxation process whereas the Kelvin-Voigt model is used to describe the creep model [8, 19]. The viscoelastic properties of the polymers are often described using a more complex four-parameter model – a Burgers model. Strain vs. time relationship for the model was defined [8] as:

$$\varepsilon = \frac{\sigma_0}{E_1} + \frac{\sigma_0}{\eta_1} \cdot t + \frac{\sigma_0}{E_2} \cdot \left( 1 - e^{-\frac{E_2}{\eta_2} t} \right) \quad (4)$$

where:  $\varepsilon$  – strain,  $t$  – time,  $\sigma_0$  – applied stress,  $E_1$  – Young's modulus in Maxwell model,  $E_2$  – Young's modulus in Kelvin-Voigt model,  $\eta_1$  – dynamic viscosity in Maxwell model,  $\eta_2$  – dynamic viscosity in Kelvin-Voigt model. Based on the creep-relaxation curve for the static force of 200 mN (the stress applied to the tested specimen being 0.24 MPa), the obtained experimental data were described using Equation 4 resulting in a very good fit ( $R^2 = 0.957$ ). Based on the determined correlation coefficients, the Burgers model parameters were calculated:  $E_1 = 6.05$  MPa,  $E_2 = 0.90$  MPa,  $\eta_1 = 9471$  MPa·s,  $\eta_2 = 32.3$  MPa·s.

## 4. Summary

The tests enabled the thermomechanical properties of HTPB-based propellant using DMA measurements in two holders to be determined: dual-cantilever and compression type. The relationships between the storage modulus and the loss modulus and between the mechanical loss modulus and the temperature and vibration frequency were determined. The results enabled the glass transition temperature of hard and soft segments in the tested propellant (–6 and –64 °C, respectively) to be determined. A change in vibration frequency shifts the DMA curves and allows the apparent glass transition activation energy of soft and hard segments in the propellant (160 and 51 kJ/mol, respectively) to be determined. The coefficient of thermal expansion was determined at  $(51 \pm 4) \cdot 10^{-6}$  1/K (from –85.0 to 95.0 °C). The effects of dynamic and static force on specimen strain were determined. Creep-relaxation measurements were used to determine a linear area of the response to static forces from 25 to 1000 mN. The creep curve was described using a four-parameter model which showed a very good fit and which was used to determine Young's modulus in the Maxwell and Kelvin-Voigt model as well as the coefficient of dynamic viscosity in that model.

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