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Influence of measurement parameters on the test results of thermomechanical properties of solid rocket propellants
Wpływ parametrów pomiarowych na wyniki badań właściwości termomechanicznych stałych paliw rakietowych

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Abstract: *The paper presents an analysis of the thermomechanical properties of a heterogeneous solid rocket propellant using dynamic mechanical analysis. Measurement conditions were optimised by varying the frequency and amplitude of the vibrations and the thickness of the sample being deformed. It was found that changing the vibration frequency affects the temperature-dependent shift of the DMA curves as well as changes in the intensity of the peaks. The greatest effect of frequency is seen in the glass transition temperatures of the soft and hard segments. This allows the material to be characterised by calculating the apparent activation energy of the individual segments. An important issue in the comparative analysis of the mechanical properties of propellants includes the measurement parameters and dimensions of the test sample. It was verified that the parameters proposed in STANAG 4540, namely a frequency of 1 Hz, an amplitude of 20 µm and a sample thickness of 2.0 mm, should be taken as the optimum conditions. DMA analysis provides highly accurate results over a wide temperature range and provides a great deal of information about the propellant under test at the same time.*

Streszczenie: *W pracy przedstawiono analizę właściwości termomechanicznych stałego heterogenicznego paliwa rakietowego z wykorzystaniem aparatu do dynamicznej analizy mechanicznej. Optymalizacji poddano warunki pomiarowe poprzez zmianę: częstotliwości i amplitudy drgań oraz grubości próbki poddawanej deformacji. Stwierdzono, że zmiana częstotliwości drgań wpływa na przesunięcie krzywych DMA w zależności od temperatury oraz zmienia się intensywność pików. Największy wpływ częstotliwości widoczny jest dla temperatur zeszklenia miękkich i twardych segmentów. Pozwala to na scharakteryzowanie materiału poprzez obliczenie wartości pozornej energii aktywacji poszczególnych segmentów. Istotnym*

zagadnieniem w analizie porównawczej właściwości mechanicznych paliw są parametry pomiaru i wymiary badanej próbki. Sprawdzono, że jako warunki optymalne należy przyjmować parametry zaproponowane w STANAG 4540: częstotliwość 1 Hz, amplitudę 20 µm i grubość próbki 2,0 mm. Analiza DMA zapewnia bardzo dokładne wyniki w szerokim zakresie temperatur oraz jednorazowo dostarcza bardzo dużo informacji na temat badanego paliwa.

Keywords: *DMA, heterogeneous propellants, glass transition temperature, apparent activation energy, STANAG 4540*
Słowa kluczowe: *DMA, paliwa heterogeniczne, temperatura zeszklenia, pozorna energia aktywacji, STANAG 4540*

1. Introduction

Solid heterogeneous rocket propellants have a wide range of applications in the defence industry. Because of their use, they are required to have properties which ensure safe use while meeting operational requirements. Such properties can be, for example, chemical stability, high heat of combustion, low sensitivity to ignition and detonation, or mechanical properties. With solid heterogeneous rocket propellants, a number of factors can affect their mechanical properties. The properties of the polymer used as a binder, the type and amount of oxidiser or crosslinking agent used, for example, will have an impact. The mechanical properties of materials determine, among other things, a material's elasticity at a given temperature, tensile properties, stiffness, ductility. The propellant in a rocket engine must be able to withstand the overloads created during flight, so it is very important to determine its mechanical properties.

Dynamic mechanical analysis (DMA) testing involves subjecting an appropriately sized sample, to oscillatory deformation. Isothermal and non-isothermal testing is possible. The method specified allows the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics or metals to be measured. In this type of analyser, the material sample is deformed in various ways e.g. by bending, stretching, shearing, compression, in order to subject it to periodic or constant stresses. Oscillatory stresses can be induced in the material as stress or strain forces, with a given force acting on the sample. A modulus which is a function of time or temperature is measured, providing information on phase transitions. An additional advantage is that the material can be described across a wide range of stiffness and/or frequency.

Depending on the properties of the material, DMA can be used to measure viscosity or elasticity. DMA measures such parameters as the storage modulus (E'), the loss modulus (E''), which are the elastic and viscous components respectively, and the mechanical loss coefficient ($tan\delta$, which is the ratio of the loss modulus to the storage modulus) [1]. The mechanical loss coefficient, or damping, provides information about a material's ability to absorb energy. Both, moduli and damping change, with the state of the material, its temperature and the frequency of stresses applied to the sample. DMA is also an excellent tool for determining phase transition temperatures, such as the glass transition temperature (T_g) of a material [2]. DMA can also be used to study the kinetics of curing, typically to determine the glass transition point and gel point of thermosets [3]. The holders in which a sample can be tested vary according to the physical state of the objects to be tested (solid, liquid), their shape (cuboid, disc, cylinder), and the type of test being performed (compression, tension, crushing).

When the mechanical properties of heterogeneous solid rocket propellants are tested using DMA, the behaviour of the rocket propellant during operation, transport or combustion in the rocket engine chamber can be assessed. The mechanical properties will change as the components of such a propellant change. Tests were carried out to determine the thermo-mechanical properties of hydroxyl-terminated polybutadiene (HTPB)-based solid propellants using DMA measurements in two holders: dual cantilever and compression [4]. The paper identifies the relationships between E', E'' and $tan\delta$ and temperature and vibration frequency. The glass transition temperatures of the hard and soft segments were determined to

be –6 and –64 °C, respectively, with the apparent glass transition activation energy of the hard and soft segments in the propellant, being 51 and 160 kJ/mol, respectively. The thermal expansion coefficient of the tested propellant was $(51 \pm 4) \cdot 10^{-6}$ 1/K.

In their paper, Cerri *et al.* [5], presented DMA measurements for a propellant sample AV04, which was formed using 8 μm diameter aluminium, representing 12% of the propellant composition. *Tanδ* values decrease with ageing. On the basis of the determined glass transition temperatures of the soft and hard segments at the given vibration frequency, the apparent activation energy can be determined. The apparent activation energy was 160 ±11 kJ/mol for the soft segments, and 51 ±7 kJ/mol for the hard segments. A similar relationship between the values of the apparent activation energies of the soft and hard segments can be observed for other propellants [5].

The results for two different holders are also presented: single cantilever type and in tension [6]. The stability of the measurements at a constant stress of 0.3% and different temperatures was analysed. Stable measurements were obtained for frequencies lower than 30 Hz. It was shown that the mechanical properties of viscoelastic materials exhibit complex temperature and frequency dependencies. The principle of time-temperature superimposition makes it possible to describe this complex behaviour with a unique standard curve of a given property as a function of frequency at a given temperature. In constructing standard curves of Young's modulus, strain and stress is essential in predicting the properties of composite propellant grain from storage to firing conditions.

Wani *et al.* [7] studied HTPB-based composite rocket propellant. They found that the choice of parameters in the experiments, such as temperature, frequency, strain and stress level, is very important regarding the results obtained, as the propellant behaves differently under different conditions. In order to accurately determine the influence of these parameters on the results, they conducted a series of experiments. As the temperature increases, the storage modulus, loss modulus and *tanδ* curves, decrease with respect to frequency. Moreover, there is a correlation between increasing temperature and decreasing frequency, which can be used in the principles of time-temperature superimposition. In addition, in transient tests, they found that the relaxation modulus decreases as the strain level increases over a given time period. The graphs of the relaxation modulus as a function of time shift toward the lower end with increasing temperature, while creep susceptibility decreases with increasing stress and decreasing temperature. The value of the T_g of the composite rocket propellant increased as the heating rate increased.

Cegla *et al.* [8] studied solid heterogeneous rocket propellant containing ammonium perchlorate(VII) (AP), aluminium (Al) powder, and poly(butadiene-acrylonitrile) (PBAN). The main objective of the study was to determine the temperature range for safe operation of this propellant. For this purpose they determined, among other things, the T_g, which was –57.1 °C (determined in accordance with STANAG 4540).

The purpose of this study was to analyse the results of thermomechanical properties using DMA. A propellant yielding a reduced amount of hydrogen chloride in the combustion products was selected for the study. The thermo-mechanical properties of the classical composition of AP/Al/HTPB-based solid heterogeneous rocket propellants are widely described in literature, while test results for modified compositions are lacking. Optimisation of the DMA measurement conditions was carried out by varying the frequency and amplitude of the vibrations and the thickness of the sample being deformed.

2. Experimental part

2.1. Heterogeneous solid propellants used for testing

Solid heterogeneous rocket propellant of the composition shown in Table 1 was used for the testing. The propellant components were mixed in a NETZSCH type PML1 planetary mixer using the classic method.

Table 1. Composition of the test propellant S1

Element	Content [wt.%]	Type and manufacturer
HTPB (R45M)	8.10	Island Pyrochemical Industries
Lecithin	0.10	Sigma-Aldrich
Dimeryl diisocyanate	1.72	Island Pyrochemical Industries
Al	12.00	Benda-Lutz
AP (coarse)	21.28	Island Pyrochemical Industries
AP (fine)	26.60	Island Pyrochemical Industries
$NaNO_3$	26.20	VWR Chemicals
nano-Fe_2O_3	1.00	Military University of Technology
Catocene	3.00	Neo Organics

2.2. Testing methods

2.2.1. DMA

A dynamic mechanical analyser, NETZSCH's DMA 242 E Artemis, was used to test the thermomechanical properties. The analyser is capable of measuring a maximum force of 24 N (combining 12 N dynamic and 12 N static force). All propellants were tested using a double cantilever type holder. Samples were prepared as 50.0×10.0 mm cuboids of thickness 2.0, 2.5, and 3.0 mm). The purpose of the holders used was to immobilise the ends of the rectangular specimen, while the centre is pressed against the moving part of the system, which generates an oscillating force. Three areas of deformation occur during measurement: expansion, shear and compression. The positioning of the sample in the double cantilever type holder is shown in Figure 1. DMA was carried out in the temperature range –120 to 105 °C with a temperature rise rate of 2 °C/min and a nitrogen flow rate of 50 mL/min. The sample was exposed to deformations at an oscillation frequency of f = {0.5, 1, 2, 5 and 10} Hz and amplitudes of 10, 20 and 30 μm.

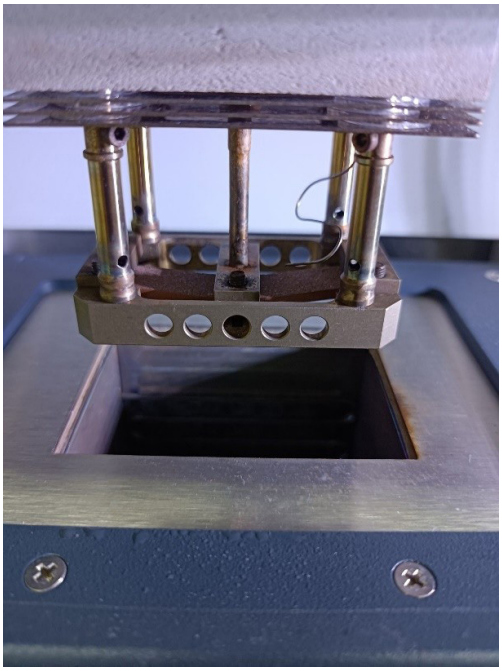

Figure 1. Double cantilever type holder with positioned test specimen

3. Results and discussion of the results

3.1. Influence of frequency on the mechanical properties of propellants

The first variable parameter investigated was the vibration frequency. Five different values were used in the measurements: 0.5, 1, 2, 5 and 10 Hz. Figures 2-4 show the temperature dependence of the storage modulus (*E'*), the loss modulus (*E''*) and the mechanical loss factor (*tanδ*) for the selected frequencies. The graphs are typical of HTPB-based solid heterogeneous propellants [7, 9-11]. The glass transition is kinetic in nature and is therefore strongly dependent on the rate of temperature increase and the vibration frequency [12]. The highest values of the storage modulus are at negative temperatures. Then, as the temperature increases, the modulus decreases and above –20 °C it stabilises at less than 80 MPa. A decrease in the value of the modulus means a decrease in the material›s ability to store energy. As the frequency increases, the value of the modulus increases. From the relationship $E' = f(T)$ the T_g can be determined as the onset of the curve. Table 2 summarises the values of the storage modulus at –115 and 25 °C and the determined T_g at different vibration frequencies.

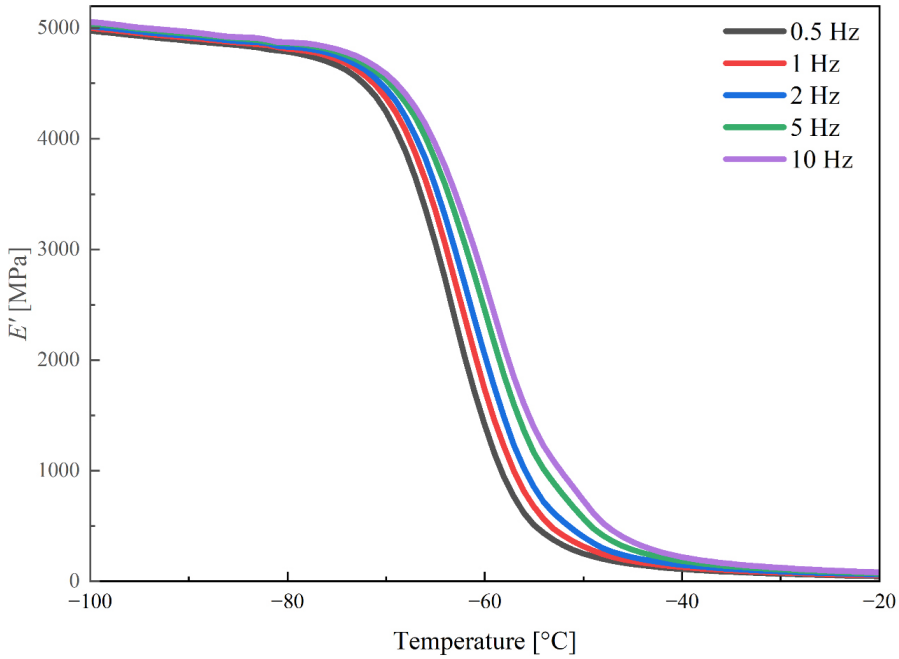

Figure 2. Temperature dependence of the storage modulus for different vibration frequencies for sample S1

Table 2. Behavioural modulus values at –115 and 25 °C and T_g for different vibration frequencies for sample S1

f [Hz]	T_{onset} [°C]	*E'* (–115 °C) [MPa]	*E'* (25 °C) [MPa]
0.5	–69.8	5178	10
1	–69.1	5204	12
2	–68.5	5220	14
5	–67.7	5246	18
10	–67.0	5266	22

The onset temperature resulting from the increasing vibration frequency increases from –69.8 to –67.0 °C, as does the behavioural modulus, at –115 °C it increases from 5178 to 5266 MPa and at 25 °C from 10 to 22 MPa.

Figure 3 shows the temperature dependence of the loss modulus at different vibration frequencies. The $E'' = f(T)$ curves show one clear peak resulting from the transformation taking place in the test sample. It can be seen that the peak is smaller and shifted towards higher temperatures as the frequency at which the sample is tested, increases. The T_g, which was determined as a maximum, shifts towards higher temperatures from –62 to –58 °C. From this peak, the T_g of the propellant is determined at a frequency of 1 Hz, in accordance with STANAG 4540 [14]. Table 3 summarises the values of the peak maximum temperature (T_g) and the loss modulus at the peak maximum at the different vibration frequencies.

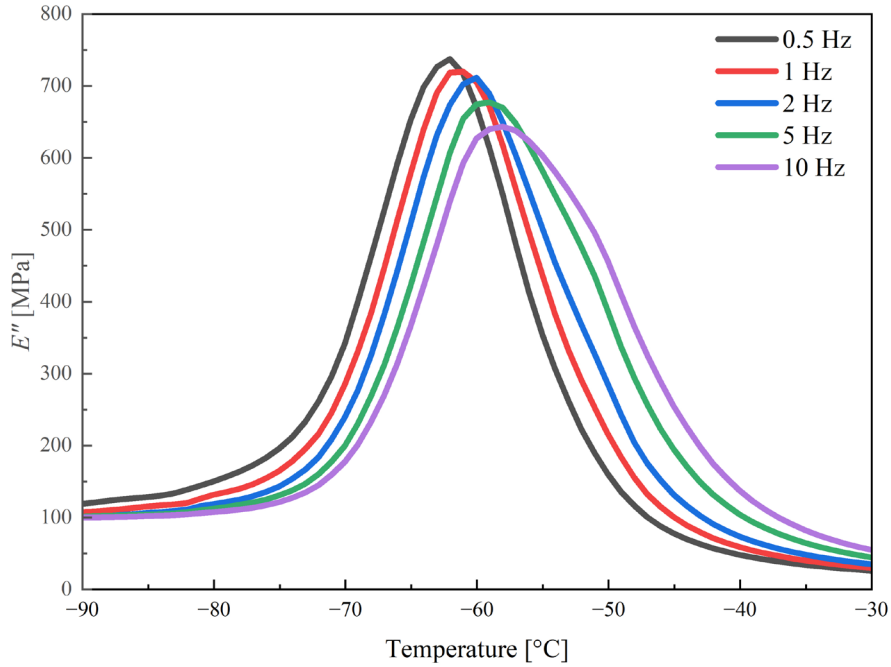

Figure 3. Temperature dependence of the loss modulus for different vibration frequencies for sample S1

Table 3. Loss modulus values at peak maximum and maximum temperature (glass transition) at different vibration frequencies for sample S1

f **[Hz]**	T_{max} [°C]	***E”*** **[MPa]**
0.5	–62.2	739
1	–61.4	725
2	–60.4	713
5	–59.3	681
10	–58.4	645

From the temperature, two peaks can be observed on the *tanδ* curve; one much higher and narrower than the other located at the higher temperatures (Figure 4). These are derived from changes occurring sequentially in the soft and hard propellant segments. The outermost segments of the HTPB main polymer chain are responsible for the soft segments in the propellant structure. Restricted mobility areas and polyurethane

groups, among others, are responsible for the hard segments in the propellant structure. The presence of particulates, and the interactions between the particulates and the binder, result in reduced mobility in the propellant structure. Table 4 summarises the glass transition temperatures of the soft and hard segments and the *tanδ* values at maximum peak for the soft and hard segments. The *tanδ* and T_g values increase for the soft segments of the propellant and decrease for the hard segments as the frequency increases.

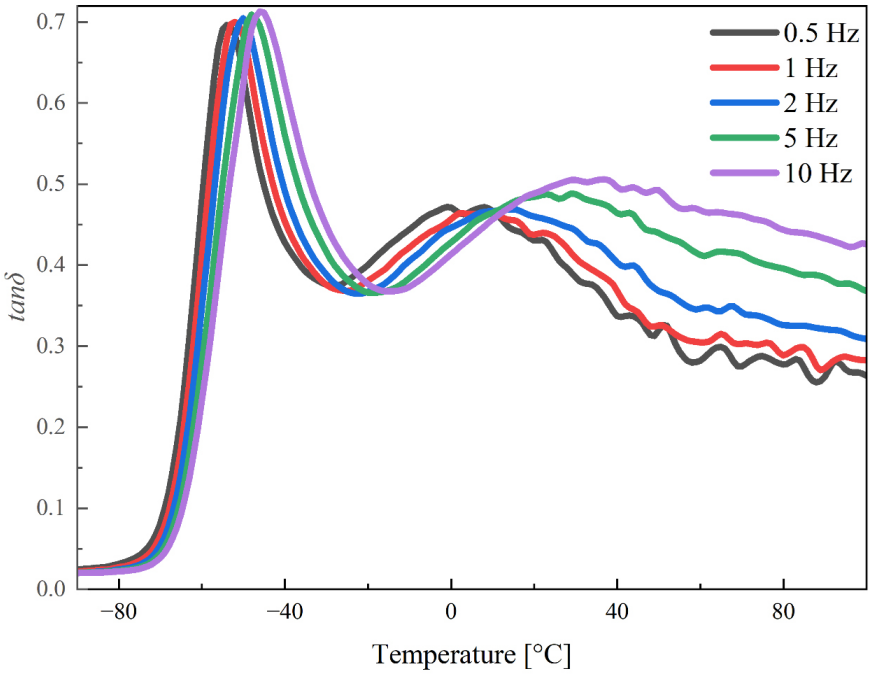

Figure 4. Temperature dependence of the mechanical loss factor for different vibration frequencies for specimen S1

Table 4. Soft and hard segment glass transition temperatures and *tanδ* values at peak maximum for soft and hard segments for sample S1

f [Hz]	T_{soft} [°C]	$tan\delta_{soft}$	T_{hard} [°C]	$tan\delta_{hard}$
0.5	–53.9	0.699	0.7	0.472
1	–52.3	0.700	9.5	0.465
2	–50.1	0.705	14.9	0.469
5	–48.0	0.709	23.9	0.489
10	–45.7	0.714	32.4	0.506

The T_g values for soft and hard segments as a result of frequency, can be described by the equation:

$$f = f_0 \cdot exp\left(\frac{E_a^a}{R \cdot T}\right) \tag{1}$$

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

Parameter calculations using Equation 1 were carried out for soft and hard segments. The calculated average values of the test propellant based on the two samples are summarised in Table 5.

Table 5. Apparent activation energies and natural logarithms from the pre-exponential frequency factor for soft and hard segments calculated for propellant S1 based on DMA measurements

Parameter	Soft segments	Hard segments
lnf_0	85.2 ±3.5	30.1 ±1.6
E_a^a [kJ/mol]	156.6 ±6.6	70.3 ±3.6

The apparent activation energy of the hard segments is lower than for the soft segments. A similar relationship can be observed for other HTPB-based propellants [10, 11, 15]. The apparent activation energy of the soft and hard segments can be used to assess the interaction of the propellant components.

3.2. Influence of vibration amplitude on the mechanical properties of propellants

The next measurement parameter to be was altered was the amplitude of the sample vibration. Tests were carried out for 10, 20 and 30 µm. Figures 5-8 show the effect of vibration amplitude on the T_g determined from the *E'*, *E"* and *tanδ.* curves. Since increasing the amplitude of vibration increases the force required to deform the sample, it is important to check that the measurement parameters set are achieved during the measurement. When a vibration amplitude of 30 µm was used, the maximum force used during the measurement was 7 N, which is not beyond the measurement capabilities of the analyser. An additional indicator is that the recorded vibration amplitude during the measurement corresponded to the value set. It was observed that the T_g shifts towards higher temperatures as the amplitude of vibration decreases (irrespective of the type of DMA curve).

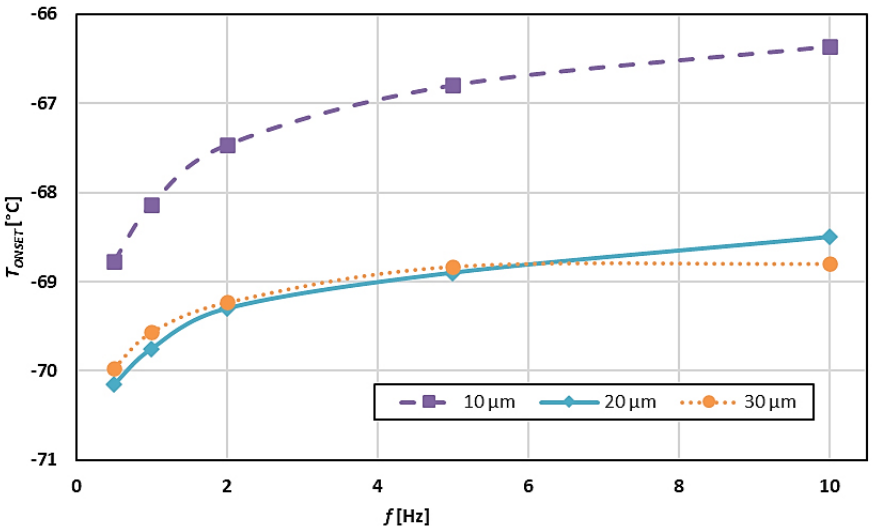

Figure 5. Effect of amplitude on T_g determined from the curve $E' = f(T)$

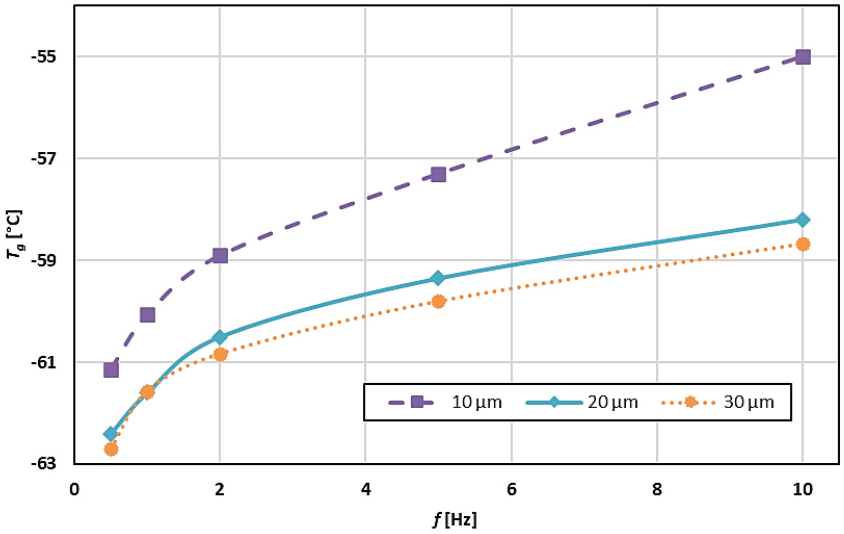

Figure 6. Effect of amplitude on T_g determined from the curve $E'' = f(T)$

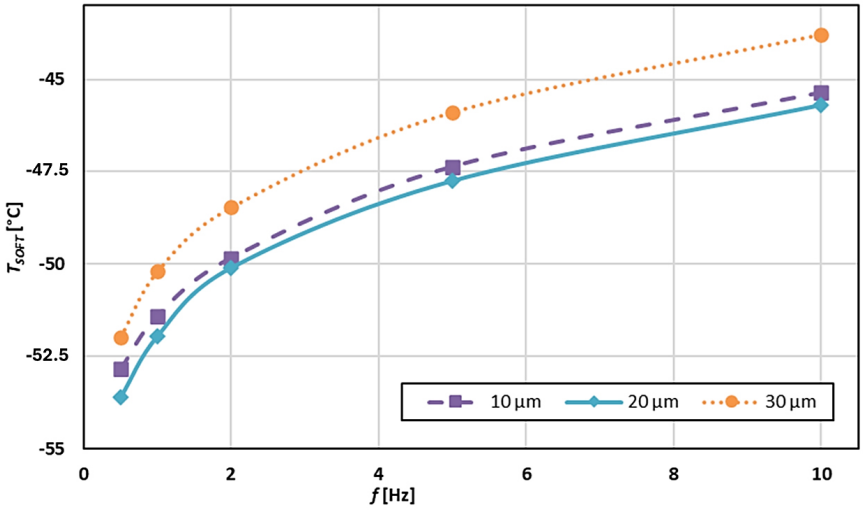

Figure 7. Effect of amplitude on T_g of soft fragments determined from the curve $tan\delta = f(T)$

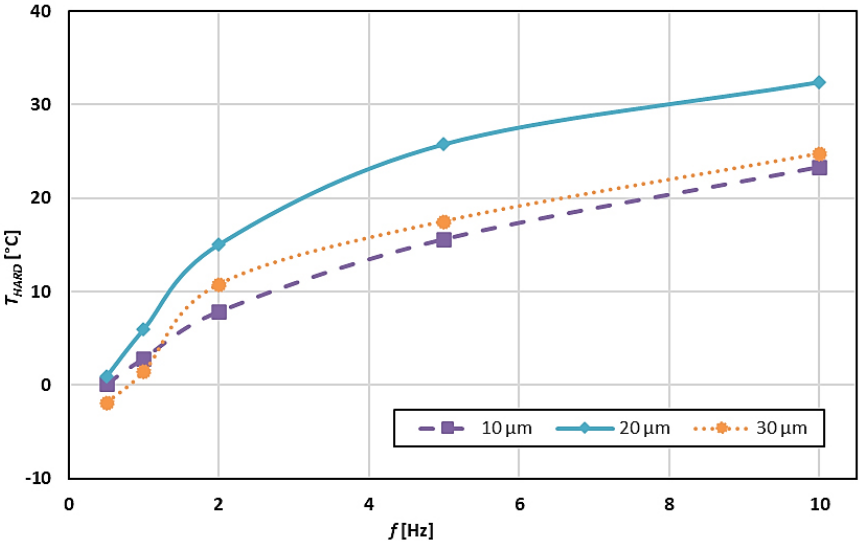

Figure 8. Effect of amplitude on T_g of hard fragments determined from the curve $tan\delta$=f(T)

Parameter calculations from Equation 1 were carried out for soft and hard segments. The calculated average values of the test propellant based on the tested samples are summarised in Table 6.

Table 6. Apparent activation energies and natural logarithms from the pre-exponential frequency factor for soft and hard segments calculated for propellant S1 based on DMA measurements

Amplitude [µm]	Parameter	Soft segments	Hard segments
10	lnf_0	90.6 ±5.3	32.9 ±2.9
	E_a^a [kJ/mol]	167.0 ±9.5	75.3 ±6.0
20	lnf_0	85.2 ±3.5	30.1 ±1.6
	E_a^a [kJ/mol]	156.6 ±6.6	70.3 ±3.6
30	lnf_0	85.2 ±3.4	31.6 ±3.0
	E_a^a [kJ/mol]	158.0 ±6.5	72.6 ±7.0

Based on measurements of the effect of vibration amplitude, its influence on T_g values was observed. It is therefore important to use the same amplitude value when comparatively testing different propellant samples. In contrast, vibration amplitude does not affect the apparent activation energy values of the soft and hard segments (the values are similar taking standard deviation into account).

3.3. Effect of sample thickness on the mechanical properties of propellants

Figures 9-12 show the dependence of test sample thickness on the T_g, determined from the curves E', E'' and $tan\delta$. As with the change in vibration amplitude, the change in thickness of the test sample directly affects the value of the force used during measurement. In the testing of a 3.0 mm thick sample, the maximum force used during the measurement was 8 N, which is not beyond the measuring capacity of the analyser used. An additional indicator is that the recorded vibration amplitude during measurement, corresponded to the value set. There was no gradual effect of sample thickness on the T_g values determined from the curves E' and E''. In contrast, the T_g of the soft segments decreases with increasing sample thickness whereas the T_g of the hard segments increases with increasing sample thickness. Parameter calculations from Equation 1

were carried out for soft and hard segments. The calculated average values of the test propellant based on the tested samples are summarised in Table 7.

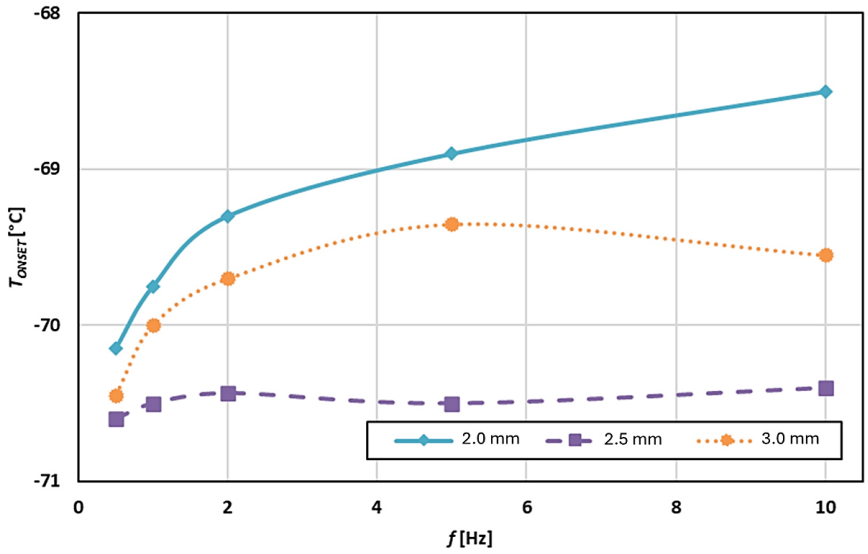

Figure 9. Effect of thickness on the T_g determined from the curve $E' = f(T)$

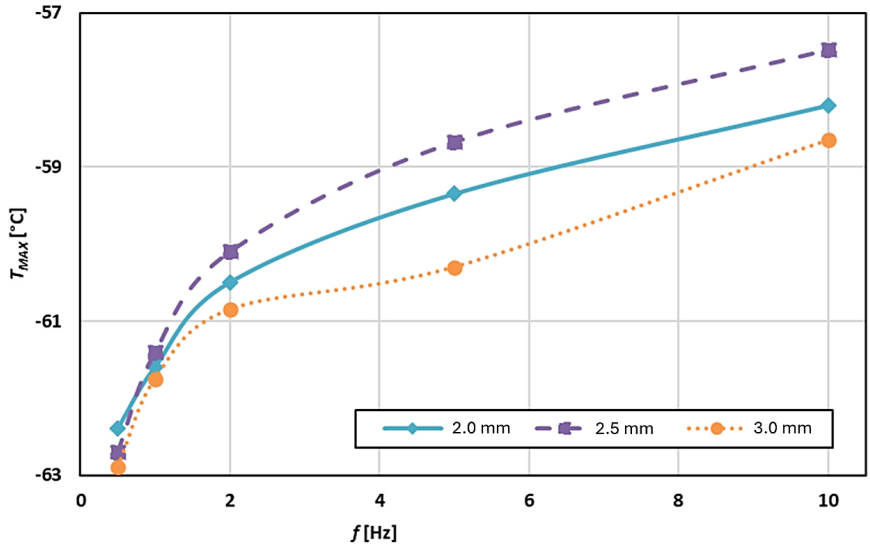

Figure 10. Effect of thickness on the T_g determined from the curve $E''=f(T)$

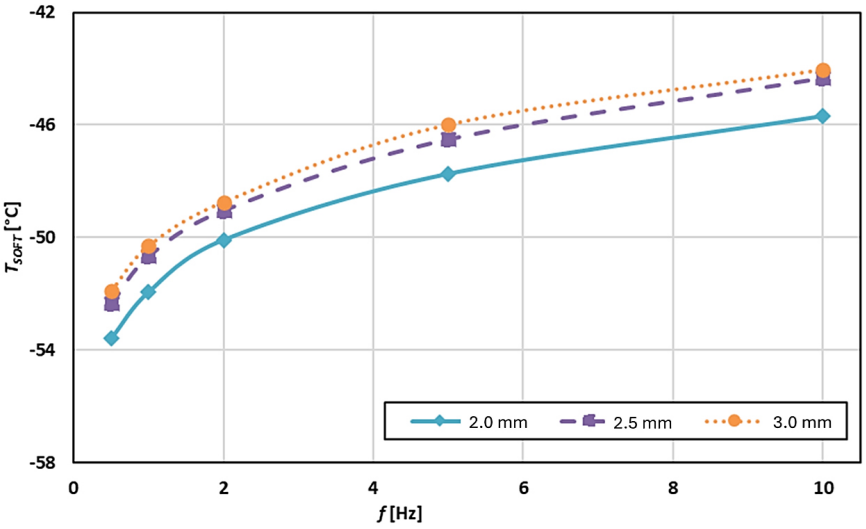

Figure 11. Effect of thickness on the T_g of soft fragments as determined by the curve $tan\delta$=f(T)

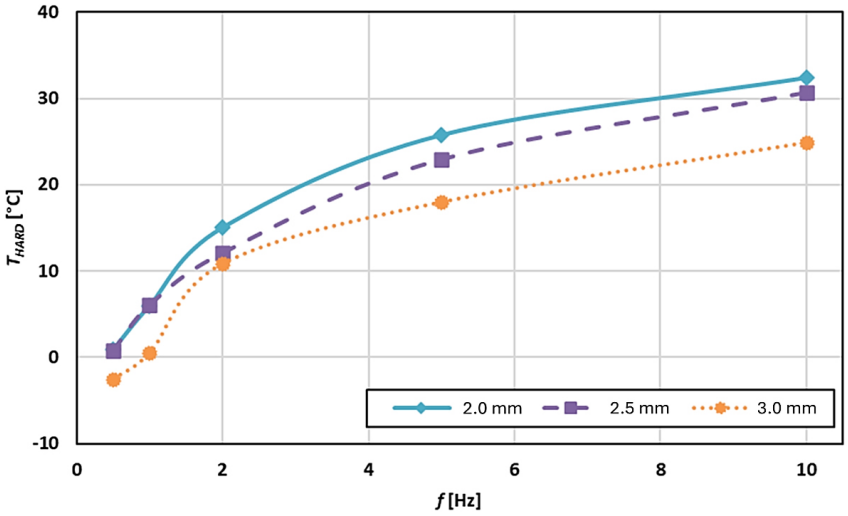

Figure 12. Effect of thickness on the T_g of hard fragments determined from the curve $tan\delta = f(T)$

Table 7. Apparent activation energies and natural logarithms from the pre-exponential frequency factor for soft and hard segments calculated for propellant S1 based on DMA measurements

Thickness [mm]	Parameter	Soft segments	Hard segments
2.0	lnf_0	85.2 ±3.5	30.1 ±1.6
	E_a^a [kJ/mol]	156.6 ±6.6	70.3 ±3.6
2.5	lnf_0	85.4 ±3.8	29.2 ±3.6
	E_a^a [kJ/mol]	157.9 ±6.7	67.9 ±8.1
3.0	lnf_0	83.4 ±7.2	33.5 ±4.2
	E_a^a [kJ/mol]	154.1 ±12.2	77.3 ±9.5

The effect of sample thickness on the T_g values was noted from measurements carried out. It is therefore important that the same thickness of propellant sample is used when comparatively testing different propellant samples. In contrast, the apparent activation energy of the soft and hard segments is similar within the error limits for different test sample thicknesses. Taking into account all the determined values of the apparent activation energies (for different vibration amplitudes and different sample thicknesses) the determined average values for the test propellant are: (158.7 ±4.9) kJ/mol for the soft segments and (72.7 ±3.7) kJ/mol for the hard segments.

4. Conclusions

- On the basis of the DMA measurements carried out, the effects of amplitude, frequency and sample thickness on the determined parameters in the DMA method for propellant with reduced hydrogen chloride in the combustion products, were determined.
- Changing the vibration frequency affects the temperature-dependent shift of the DMA curves with the intensity of the peaks changing. The differences in glass transition temperatures determined from the curve $E' = f(T)$ are approx. 2 °C, and from $E'' = f(T)$ are approx. 4 °C, depending on the frequency. The greatest effect of frequency is seen on the glass transition temperatures of the soft and hard segments, enabling the material to be further characterised by calculating the apparent activation energy of the individual segments. The effects of vibration amplitude and sample thickness on the determined glass transition temperatures are insignificant, up to 2 °C.
- Changing the measurement parameters and sample thickness does not change the apparent activation energy for the soft and hard segments. The determined mean values of the apparent activation energies for the propellant containing sodium nitrate(V) in its composition were: (158.7 ±4.9) kJ/mol for the soft segments and (72.7 ±3.7) kJ/mol for the hard segments.
- For an accurate comparative analysis of different materials, it is important to bear in mind that the measurement parameters and dimensions of the test sample affect the DMA curves. For this reason, precise parameters need to be defined to study the effect of composition on the results of thermomechanical properties with all materials being characterised in this way. The selection of measurement parameters should also take into account the capabilities of the DMA so that the parameters used are feasible during the measurement. What is key here are the force values and the vibration amplitude actually achieved during the measurements being carried out. DMA analysis provides very accurate results over a wide temperature range while providing a great deal of information about the tested propellant at the same time.

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