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ORIGINAL ARTICLE

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Investigation of thermomechanical properties of solid rocket propellant used in multi-barrel rocket systems

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Abstract The effectiveness of multi-barrel rocket systems on today's battlefields is strongly dependent on the reliability of operation and, hence, proper action of all components, especially rockets and propellants. Therefore, the properties of the solid rocket propellants used in the rocket motors must be determined with an efficient and reliable tool providing repeatable results. The article presents the results of a thermomechanical analysis of solid double-base rocket propellant used in multi-barrel rocket systems. One of the recommended methods for testing solid rocket propellants is dynamic mechanical analysis. Mechanical properties such as the dynamic storage modulus (E'), the dynamic loss modulus (E''), and the tangent $\tan(\delta)$ of the phase shift angle (E''/E') were measured with the use of the TA Instruments DMA Q 800 device, in a temperature range of $-$ 100 to +100 °C with the use of different frequencies of applied force and heating rates. Special attention was devoted to determining the glass transition temperature following the STANAG 4540 standardization agreement, as well as the influence of testing parameters on the obtained experimental results. Dynamic mechanical analysis has proven to be an effective method for the evaluation of key properties influencing rocket motor behavior.

Keywords Dynamic mechanical analysis · Solid rocket propellants · Glass transition temperature

1 Introduction

The modern battlefield is saturated with equipment specialized in destroying armored vehicles, critical infrastructure, and fighting enemy manpower. Anti-tank weapons equipped with HEAT and APFSDS warheads are used for precise point attacks against enemy vehicles such as tanks and armored personnel carriers [1,2]. Weapons that combine long-range precision strike capabilities with an area-of-effect attack focused on destroying everything in its path are barrel and rocket artillery [3–5]. Modern multi-barrel rocket artillery systems are the weapon of choice for both regular armies of today's world superpowers and guerrillas and terrorist groups in various armed conflicts. The most recognized weapon system in this family is the US Army's MLRS—Multiple Launch Rocket System, which is a tracked vehicle, and HIMARS—High Mobility Artillery Rocket System, a wheeled version based on the same 227-mm rocket. On the other side of the front, the most recognized is the 122-mm BM-21 GRAD high-mobility wheeled vehicle. Its origins date back to the 1960s, and it has been adopted by different countries and undergone upgrades since then [6–8]. The main advantage of multi-barrel

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rocket systems is to deliver a devastating, accurate artillery bombardment in a short period of time, covering a relatively large area. The other advantages are high mobility depending on the type, fast deployment ability, and relatively easy handling and operation compared to traditional howitzers. Their disadvantage is the possibility to determine their position after firing, due to the relatively large smoke signature connected to the rapid firing of multiple rockets [7,8]. For the successful operation of these weapon systems, especially their survivability on today's battlefields, they must be able to be prepared, carry out the fire mission, and escape as quickly as possible in order to avoid being destroyed by enemy artillery counterfire or other means.

In order to achieve the mentioned effectiveness in mobility and operation, the munitions used in the weapon system must meet high safety standards. One of the key characteristics of the solid rocket propellant, of which the characteristics are similar to those of general polymers, is the glass transition temperature. During the transition of the polymeric material from the elastic rubbery state to the hard brittle glassy state, cracks and voids may occur in the material. These cracks can lead to an uncontrolled increase in the burning surface of the propellant grain and, hence, an increase in the pressure in the motor chamber during firing and, in the worst case, an explosion of the rocket [9–11]. In peace conditions, rocket motors are especially subjected to natural aging during storage for long periods of time [12]. In order to simulate the aging of the propellants and predict the possible impact of prolonged storage on mechanical properties, artificial aging procedures are applied. This issue is described in detail in the literature [13–16]. Thermomechanical parameters of the propellant, particularly the glass transition temperature, must be determined with the most sufficient and effective method, ensuring reliable and repeatable results. Among thermo-analytical techniques suitable for solid rocket propellant testing and determination of the glass transition temperature, the most widely used in the relevant literature include Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA); however, NATO Standardization Agreement STANAG 4540 points to DMA as the more recommended method [17,18]. In previous work [19,20], authors have conducted research on the determination of the glass transition temperature of solid rocket propellants with the use of dilatometry (DIL), DSC, and DMA. In the case of the DIL technique, the glass transition of the tested propellants was determined at the onset of relative elongation curves, and their values correspond to the ones obtained in different methods; nevertheless, the onset of the curve is difficult to distinguish. In the case of the DSC technique, the glass transition temperature is determined based on changes in the specific heat value on the heat–temperature–dependence curve. These changes are, however, very insignificant, and it would be nearly impossible to determine the glass transition temperature of selected propellants without knowing the indicative area of search [19]. The mentioned problems do not occur in the case of DMA experiments as the glass transition in energetic materials is strongly related to the change in their mechanical, rather than thermal, properties. The glass transition temperature of solid rocket propellants, tested with the use of DMA, occurs within a region defined by relatively easy-to-distinguish points [19,20]. DMA allows testing of the mechanical properties of solid rocket propellants with one sample covering a wide temperature range. The method applies a periodical force of a relatively small amplitude generated by a stepper motor-driven oscillator, which causes stress that generates strain. In the case of a viscoelastic material, the properties of which are demonstrated by solid rocket propellants, the deformations are out of phase with respect to the stresses by the angle δ, and the more material there is, the less elasticity [21]. The values of the dynamic modulus of elasticity E', dynamic loss modulus E'', and damping $\tan(\delta)$ are recorded as the measurement result. The modulus E' is responsible for the elastic properties of the material, and the modulus E'' for the viscous properties. With regard to the measurement cycles, these modules are appropriately proportional to the energy stored and dissipated during the measurement. The tangent of the phase-shift angle reflects the ability of a material to dissipate mechanical energy through the rearrangement of molecules and internal friction [22,23]. The glass transition temperature of solid rocket propellants, determined by the DMA method, is a particularly useful parameter in determining the propellant's resistance to dynamic loads during ignition at low temperatures [24]. Literature data indicate several methods of determining the glass transition temperature based on characteristic points on the DMA curve. These temperatures include the onset of the storage modulus E' curve, the maximum of the loss modulus curve E'', and the maximum of the $\tan(\delta)$ curve. In accordance with the guidelines of the STANAG 4540 agreement [17], the glass transition temperature Tg should be determined at the maximum point of the E'' curve. The softening point of the rocket propellant determines the ability of the fuel cartridge to maintain its shape and strength. Its value can be determined using the maximum of the $\tan(\delta)$ curve in the positive temperature range on the Celsius scale. Thus, the DMA method is a tool enabling the determination of the temperature range of solid rocket propellant exploitation [17]. The DMA measurements provide valuable information for the development and testing phase of solid rocket propellants and enable the characterization of their viscoelastic properties. The mechanical properties of propellants measured with the DMA technique allow evaluation of their behavior during operation within the rocket system exposed to periodic dynamic loads during transport, service, and maintenance procedures and during combustion in the rocket engine compartment [24]. A considerable safety advantage of the DMA method is the relatively small size of the samples, which do not pose a threat to the operator and the device in the case of uncontrolled ignition. The aim of the thermomechanical analysis of the propellant with the use of the DMA method was to determine the temperature range of safe operation, which can be established on the basis of glass transition and softening temperatures, and to develop an internal testing procedure for solid rocket propellant testing in accordance with the STANAG 4540 agreement.

2 Experimental procedure

2.1 Solid rocket propellants

Solid rocket propellants used in multi-barrel rocket artillery are high-energy materials with mixed flammable and oxidizing substances. The propellants have a number of properties that enable their efficient and safe usage. Chemical durability, high heat of combustion, low sensitivity to ignition and detonation, low coefficient of thermal expansion, and low molecular weight of combustion products are the key characteristics [25]. Relatively simple design, production, handling, transport, and deployment as well as their prolonged storage without degradation make them suitable for wide application [26–28]. Two general types of solid propellants can be distinguished, considering chemical content and the connection between ingredients: homogeneous (double-base) and heterogeneous (composite) solid rocket propellants. Homogeneous propellants form a uniform physical structure of chemically bonded fuel and oxidizer ingredients, whereas, in heterogeneous propellants, the oxidizer and fuel are physically mixed without creation of chemical bonds. Typical double-base rocket propellants are a mixture of nitrocellulose 50–60%, nitroglycerine 30–49% along with plasticizers, stabilizers, and burning rate catalysts [29]. Heterogeneous solid rocket propellants are a mixture of a solid oxidizer (ammonium perchlorate—AP) and combustible substances (binders), mostly synthetic rubber with functional groups, a cross-linking agent, and various kinds of plasticizers and additives [30]. Moreover, in the available literature, information can be found on composite double-base propellants modified with additives typical for heterogeneous fuels such as aluminum powder or high explosives such as HMX [31]. Both of the main groups of propellants can be applied to propel rockets in multi-barrel artillery systems. The propellant used for the experimental part of the presented research belongs to the group of homogeneous double-base propellants. The chemical composition was determined with the use of the Waters HPLC Device with a UV–VIS/PDA detector. The chemical composition of the propellant is presented in Table 1.

Samples of the rocket propellant were taken from the propulsion system of an unguided rocket. The samples were processed with a lathe with non-sparking cutting material (beryllium) and a milling head for face milling in order to achieve the desired dimensions of $60 \times 10 \times 2$ mm (length, width, and thickness). In order to ensure the proper experimental conditions for the rocket propellant, preliminary experiments were conducted for the sample of PTFE material with the same dimensions as the propellant, applying the same experimental conditions.

2.2 Method and conditions

Based on the literature data and our own experiments, DMA was chosen as the method for accurate and reliable determination of the glass transition temperature of the selected propellant. The experiments discussed in the article were conducted on the DMA Q800 device developed by TA Instruments with the use of dual-cantilever clamps. The experimental conditions are presented in Table 2.

Table 1 Rocket propellant's main chemical composition

Ingredient	Content %
Nitrocellulose	55–57
Nitroglycerine	26.3–27.7
Dinitrotoluene	8.3–10.3
Other additives	5–10

Table 2 DMA experimental conditions

Parameter	Unit	Value
Temperature range	°C	− 1
Frequency of applied force	Hz	0.1, 1 and 10
Sample conditioning time in − 100 °C	min	10
Heating rates	K/min	1, 2 and 5
Deformation amplitude	µm	20
Force resolution	N	0.00001
Strain resolution	nm	1
Modulus precision	%	± 1
Tan (δ) resolution	–	0.00001

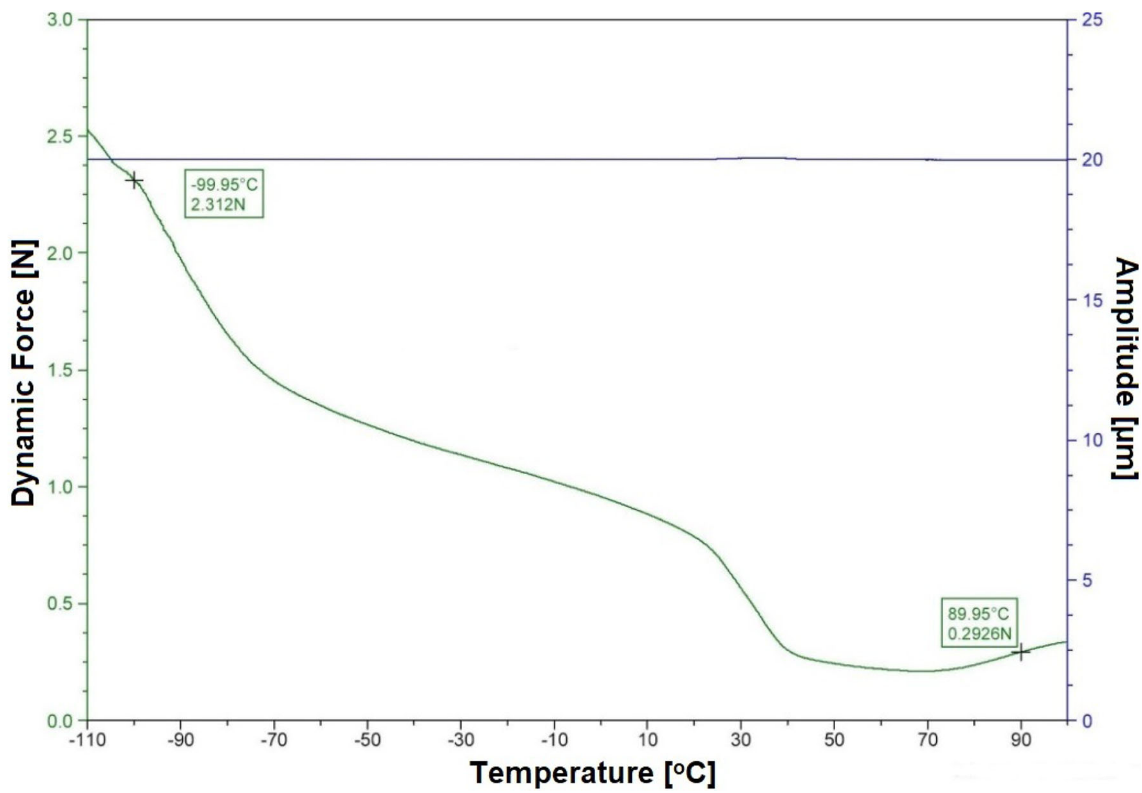

Fig. 1 DMA results for the PTFE reference sample—dynamic force and amplitude

The experiments were carried out for different frequencies of applied force and heating rates, but only one parameter was changed each time. The temperature characteristics of the dynamic storage modulus E', dynamic loss modulus E'', and tan(δ) for the propellant were determined. In order to further investigate the issues related to the temperature range of safe operation, as well as to determine the effect of the frequency of the applied force and the heating rate on the results of the DMA tests, three measurements were taken for three frequencies of 0.1, 1, and 10 Hz and three heating rates of 1, 2, and 5 K/min. The main experimental part concerning the propellant was preceded by testing the PTFE samples with similar dimensions as the propellant and under similar experimental conditions. The purpose of the test was to calibrate the device and check its operation with the use of material characterized by high thermal resistance and the ability to maintain its properties in a wide temperature range.

3 Results and discussion

The results of the DMA experiment for the PTFE reference sample are presented in Figs. 1 and 2.

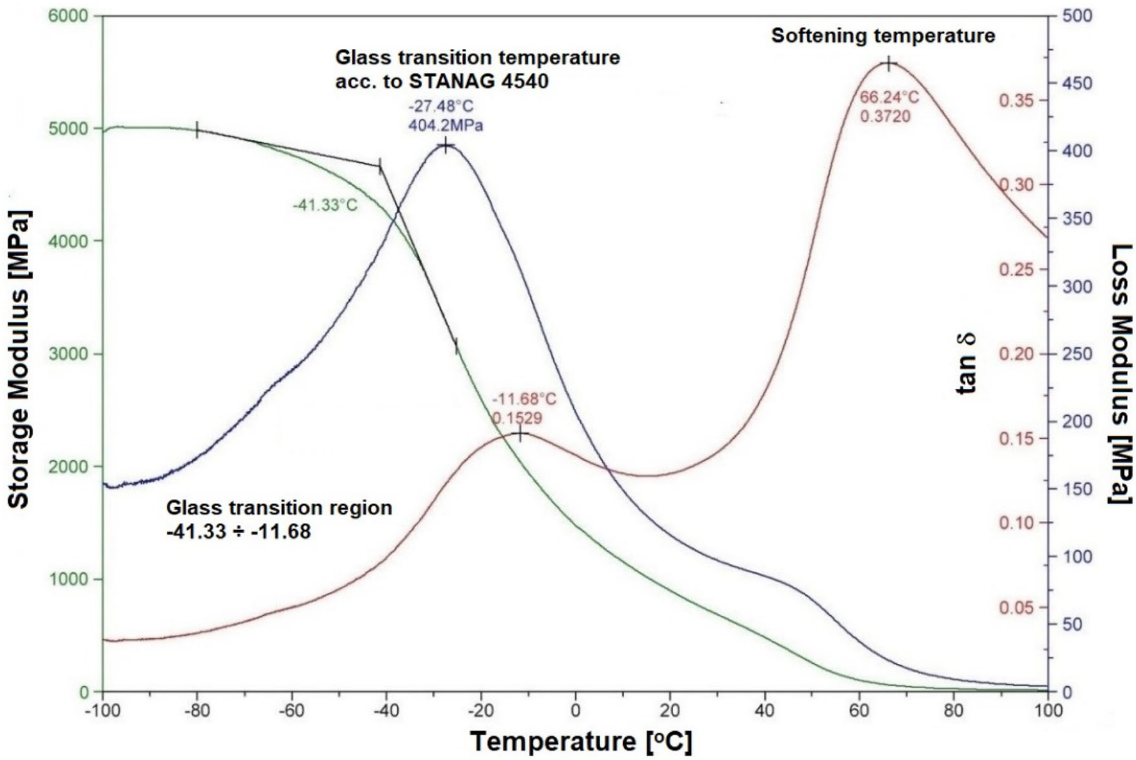

Fig. 2 DMA results for the PTFE reference sample—storage modulus (E'), loss modulus (E''), and tan(δ)

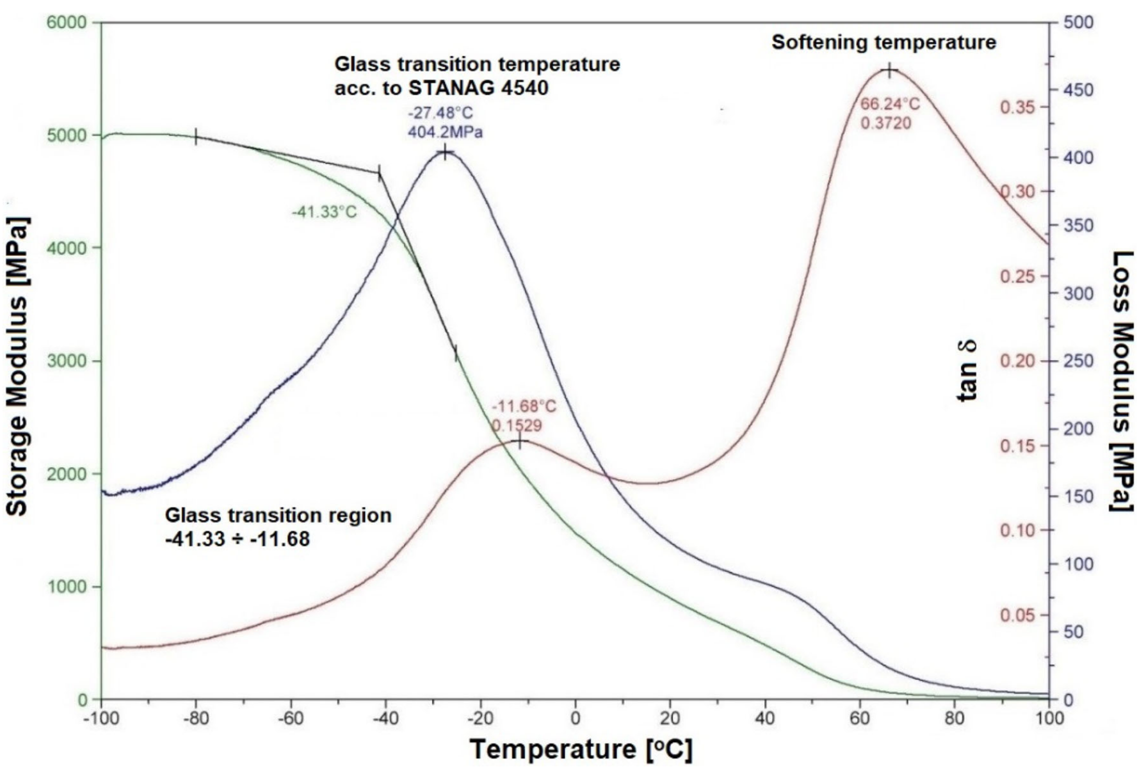

Fig. 3 DMA results for the propellant reference sample—storage modulus (E'), loss modulus (E''), and tan(δ)

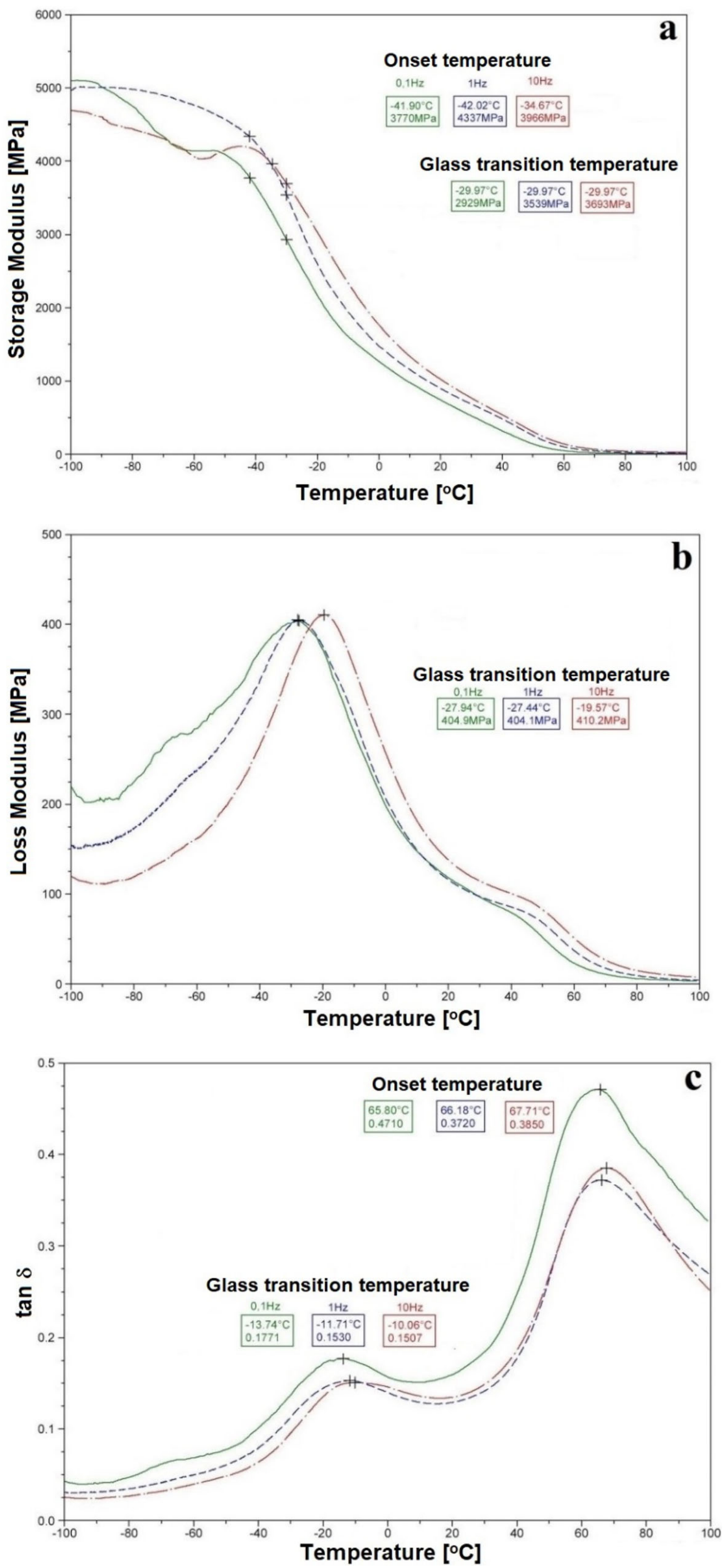

Fig. 4 DMA results for the propellant for the frequencies of 0.1, 1, and 10 Hz. **a** Storage modulus (E'), **b** loss modulus (E''), and **c** tan(δ) as function of temperature

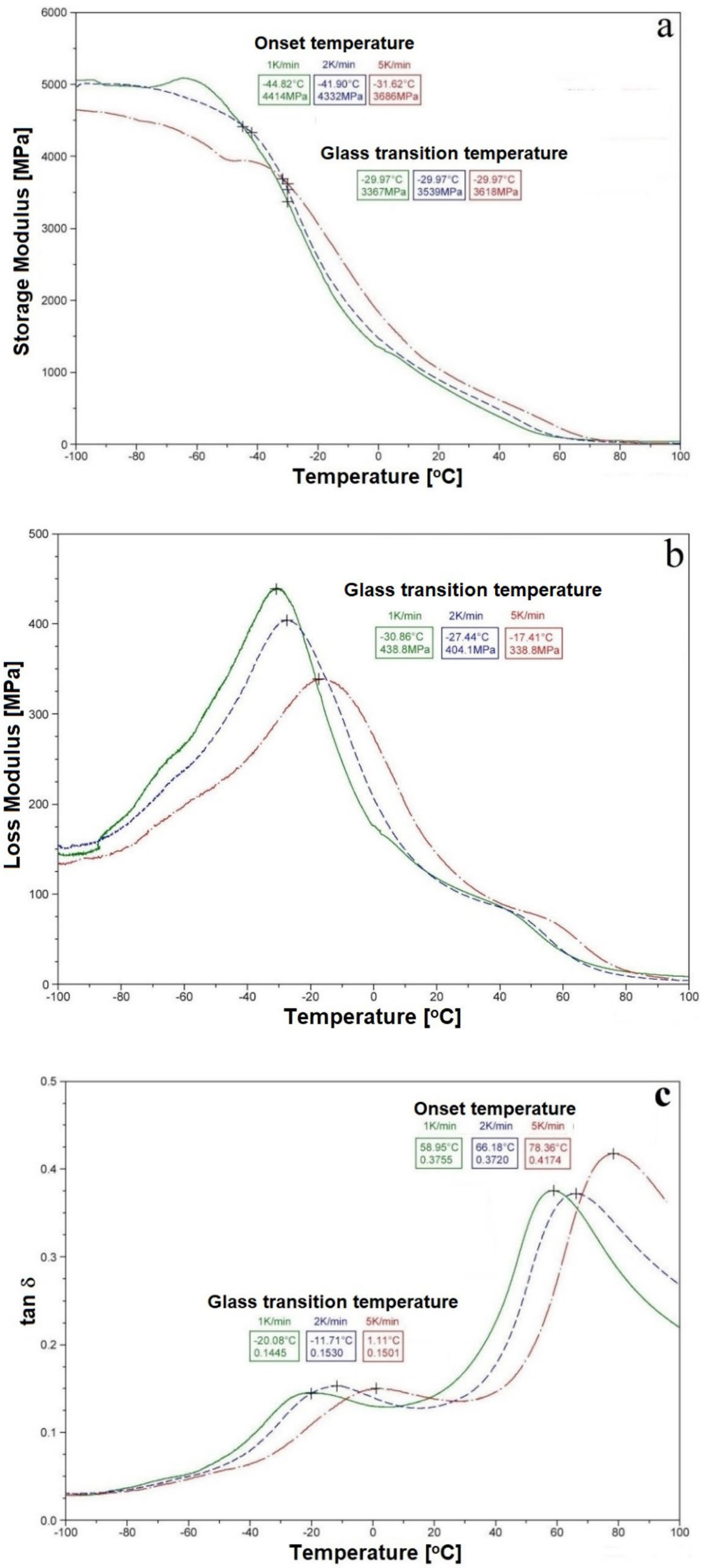

Fig. 5 DMA results for the propellant for the heating rates of 1, 2, and 5 K/min. **a** Storage modulus (E'), **b** loss modulus (E''), and **c** tan (δ) as function of temperature

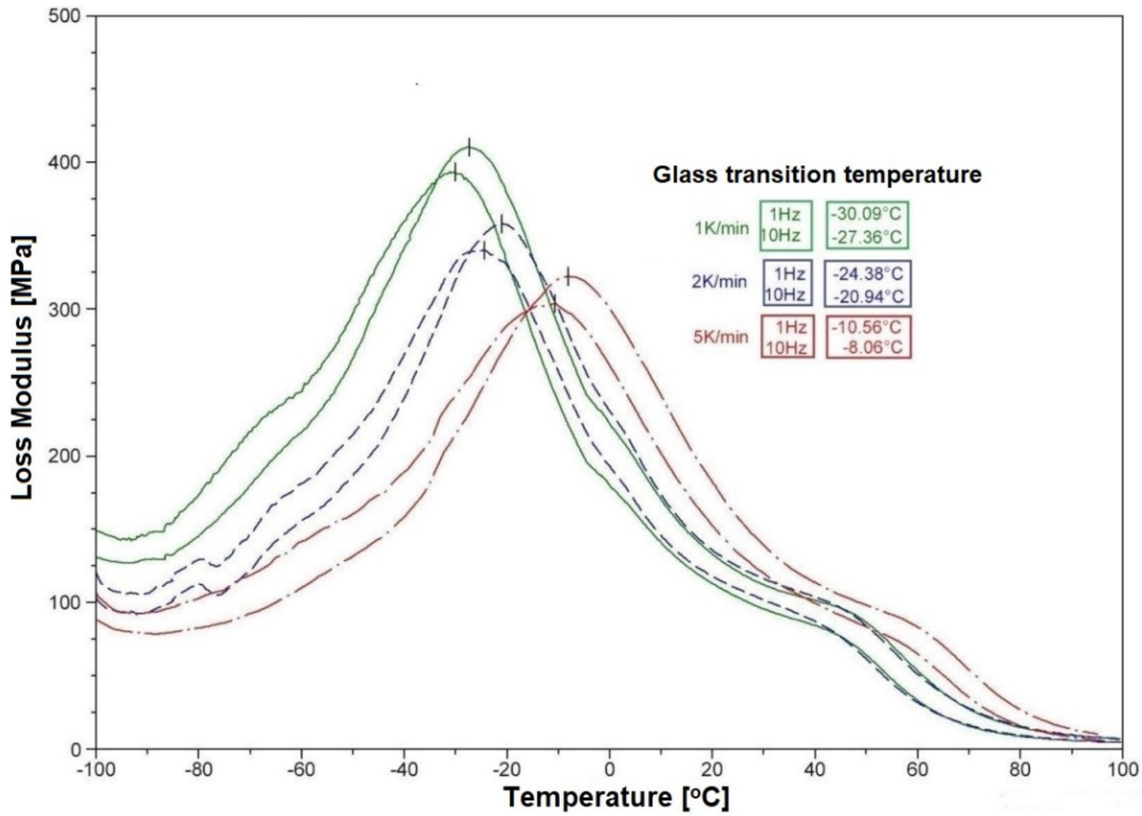

Fig. 6 Glass transition determination on the loss modulus (E'') curve for changing heating rates and frequencies

The results of the DMA analysis of the reference material are consistent with the literature data [32], on the basis of which conclusions were drawn as to the correct configuration and calibration of the DMA device. Figure 3 shows the temperature dependence of $E'$and E'' moduli and tan(δ) for the frequency of 1 Hz of the propellant.

Based on the results of the DMA tests, the temperature range of safe operation was determined. The glass transition temperature measuring − 27.48 °C, determined in accordance with the standardization agreement STANAG 4540 at the maximum of the loss modulus curve E'' for the frequency of 1 Hz, was considered as the lower-temperature limit of operation. As a result of the transition of rocket propellant into a glassy state, the stiffness of the material increases, which may lead to cracking and, consequently, to an uncontrolled increase in the combustion surface. It must be added that the glass transition of materials occurs within a region set between the onset temperature of the storage modulus curve and the peak of the tan(δ) curve in the subzero range. However, in the case of solid rocket propellant safety testing, the contractual value of the glass transition is determined at the maximum peak of the loss modulus curve. The softening temperature measuring 66.24 °C was determined at the peak of the tan(δ)g curve. After the softening point is exceeded, the propellant grain will not retain its proper shape. Exceeding both temperatures is of particular importance at the moment of ignition because of the rapid increase in the loads to which the rocket propulsion system is subjected. The results of DMA experiments with different frequencies and heating rates are shown in Figs. 4 and 5, respectively.

In the case of the dependence of the E' module on the temperature for three frequencies (Fig. 4a), an increase in the modulus value is visible with the increase in the frequency at the glass transition point (assumed on the basis of the 1-Hz frequency measurement—Fig. 3). In the vicinity of the onset point of the curve (the beginning of the glass transition region), this relationship is not maintained for the 10-Hz frequency. The possible explanation for this phenomenon is that with the increase in frequency, the material cannot deform fully in the given time period; hence, the modulus connected to the elastic properties increases. This feature is characteristic of polymer materials to which solid rocket propellants are very similar. In higher temperatures, the polymer chains move more easily, and the values of the storage modulus are similar regardless of the frequency [23]. The influence of frequency on the measurement results of glass transition and softening

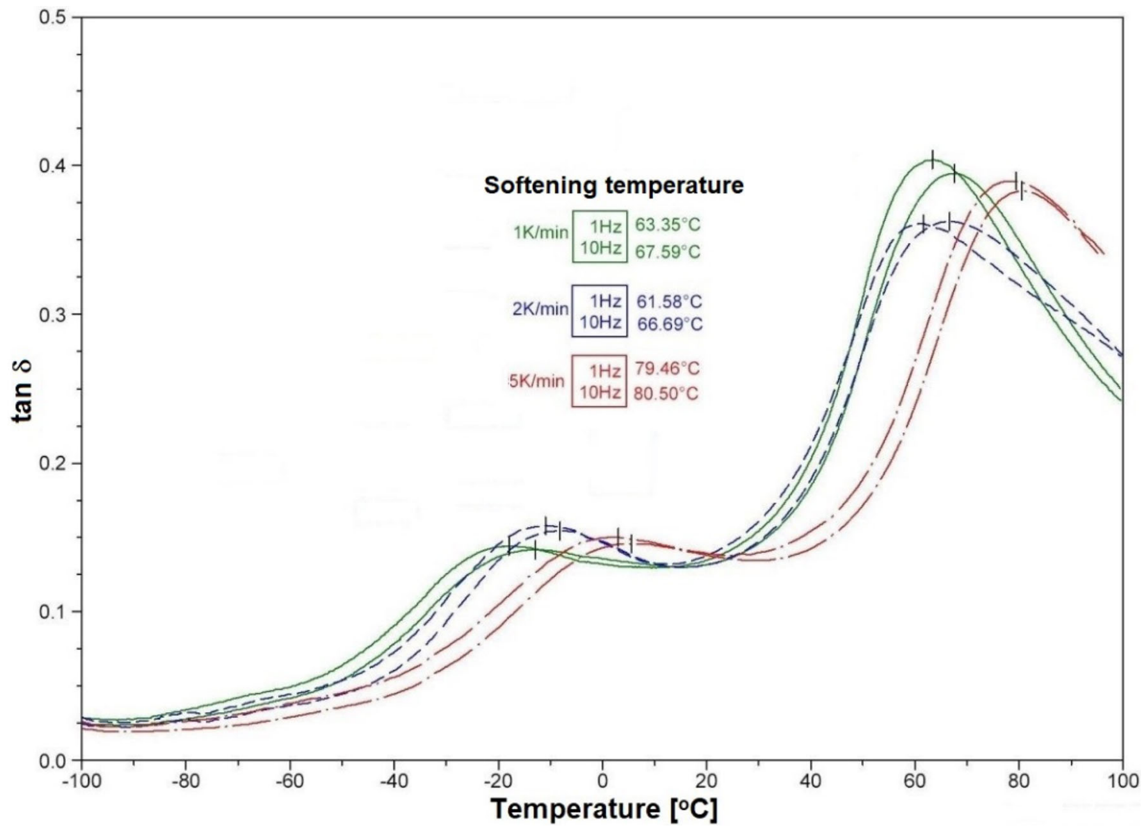

Fig. 7 Softening temperature determination on the tan (δ) curve for changing heating rates and frequencies

temperatures can be observed in the plots of the E'' and tan(δ) (Fig. 4b, c). Both temperatures increase with an increasing frequency. The values of the loss modulus are very similar for the frequencies of 0.1 and 1 Hz, and a more pronounced increase can be observed for 10 Hz. A possible cause of the increase in the value of the modules and the glass transition temperature of the tested propellant may be the fading motion of molecular chains, leading to an increase in the stiffness of the material. With the increase in the heating rate, the values of the glass transition and softening temperatures of the propellant increase and the value of the loss modulus decreases, while in the case of the dependence of the storage modulus on the heating rate, the upward trend is not maintained (Fig. 5a). Three additional tests were carried out, with different heating rates, using two frequencies simultaneously in each. The results of the measurement of the glass transition and softening temperatures on the basis of the E'' and tan(δ) curves are presented in Figs. 6 and 7, respectively.

Figure 7 shows an increase in the value of the glass transition temperature of the propellant with an increase in the frequency of the applied force and the heating rate. For each successive heating rate, a higher-temperature value for 10 Hz than for 1 Hz can be observed. The value of the loss modulus E'' increases within a single measurement and is higher for 10 Hz, while it decreases with the heating rate. The dependence of the softening point value (Fig. 7) increases within a single measurement, as was the case of the glass transition temperature, but it does not maintain an upward trend for all three heating rates. The softening point determined at the heating rate of 2 K/min is slightly lower than for 1 K/min. The obtained dependencies of the influence of the experimental conditions on the results of DMA studies are partially in line with the literature data [33–36]. Compatibility occurs in the case of the dependence of the glass transition temperature on the frequency and heating rate and the value of the storage modulus at a given temperature above the beginning of the glass transition area (Figs. 4a, 5a).

4 Conclusions

Thermomechanical analysis of solid homogeneous double-base rocket propellant used in the propulsion systems of an unguided artillery rocket system was carried out. The series of experiments were conducted by means of dynamic mechanical analysis using the DMA Q800 from TA Instruments. The glass transition and softening temperatures of the propellant were determined based on the characteristic points on the loss modulus and tan(δ) curves. On the basis of the conducted experiments, the following conclusions can be drawn:

1. The conducted DMA tests allowed us to determine the temperature range of safe operation of the tested double-base rocket propellant. The lower limit of the range is the glass transition temperature, determined at the maximum point of the loss modulus curve, based on the guidelines of the STANAG 4540 standardization agreement, while the upper softening temperature was determined at the second maximum point of the tan(δ) curve. The glass transition and softening temperatures were −27.48 °C and 66.24 °C, respectively, and were determined at a heating rate of 2 K/min, a frequency of 1 Hz, and an amplitude of 20 µm.
2. The experiments were carried out at three different frequencies of 0.1, 1, and 10 Hz and at three heating rates of 1, 2, and 5 K/min. The research carried out with the use of various parameters of the experiment allowed us to study their influence on the results of DMA tests and to conclude that increasing the frequency and heating rate causes displacement of the value of the glass transition temperature and softening toward higher temperatures.
3. The glass transition temperature of the tested propellant was determined by the DMA method following the recommendations of the STANAG 4540 standardization agreement, which specifies that the glass transition temperature of solid rocket propellants should be determined on the basis of the easily distinguishable maximum of the E'' curve. The results can therefore be used to develop other research methodologies based on the mentioned standard.

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Declarations

Conflict of interest The authors declare no conflict of interest

Author contributions Cegla M. and Czerwinska M. conceptualized the study; Cegla M. helped in methodology, writing—original draft preparation, visualization, and formal analysis; Czerwinska M. was involved in software and investigation; Cegla M., Czerwinska M., and PK validated the study; Czerwinska M., PK contributed to writing—review and editing; PK supervised the study.

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