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Methodology for Testing High-Energy Materials Under Low Temperature Conditions

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Abstract. The methodology developed for testing gun propellants at low temperatures according to PN-EN ISO 604:2006 is presented in the paper. Brief characteristics are given of the materials tested and the most important static compression test conditions, such as specimen dimensions, deformation velocity and temperature range for selected propellants, i.e. JA-2 and SC. To verify the methodology developed, preliminary strength tests were performed at selected temperatures (25, 0, -25 and -50°C). Tests were carried out on specimens fabricated by shortening the propellant grain to the dimensions required by the reference standard. The results obtained confirmed the expected strength properties for both propellants (tensile strength and brittleness). Due to its chemical composition, the JA-2 propellant is a material of low brittleness even at -50°C. It does not crack completely and only its yield point increases. The results obtained for the JA-2 propellant were consistent with those published in reference literature. The SC propellant proved to be very brittle even at room temperature. At temperatures below 0°C, it fractures completely after reaching the desired deformation.

The results obtained confirm that the adopted strength test conditions and the way the tests were prepared and performed enable acquisition of comparable and reliable results. It can be seen by analysing the results for the JA-2 propellant, which are consistent with the data in the available references. In contrast, the tests on the SC propellant proved the validity of strength tests on this type of material. Brittleness of propellant grains is a very undesirable phenomenon. A change in the combustion surface of low explosives caused by the process of propellant grain fracturing can adversely affect the magnitude and course of the pressure pulse, leading to failure of a cartridge chamber or gun barrel.

Keywords: mechanical engineering; low temperature tests; strength testing; static compression test; gun propellants

1. INTRODUCTION

For years, static strength testing of materials has been widely used to determine the mechanical properties of both classical materials, which have been used in engineering for many years and modern materials, whose properties have not yet been described in any sources. Strength testing can be carried out on a wide range of materials, i.e. highly ductile materials such as rubber, as well as brittle/fragile materials such as glass or dolomite. The data obtained from strength testing can be applied in numerical problems to determine, for example, the constants of the Johnson-Cook (J-C) constitutive equation [1]. Strength tests are also performed under various temperature conditions, both at low and high temperatures [2]. High-energy materials are a group of materials whose mechanical properties at low temperatures are of great interest to the defence industry. These include gun propellants, solid rocket propellants and explosives. Most of these materials behave as elastomers, which become brittle or fragile when exposed to low temperatures, posing a potential danger to users. A crushed high-energy material has a larger surface area during its combustion, which generates a higher combustion gas pressure. This effect may lead to failure of an artillery gun or a rocket missile and in extreme cases to the loss of life or health of the military personnel operating the ordinance. Currently, documented strength testing of gun propellants is limited to only a few selected propellants, including JA-2, M30, and XM39. Examples of such studies are not found in generally available Polish literature.

The main objective of this paper is to present a methodology for strength of gun propellants under different temperature conditions, using JA-2 and SC propellants as examples. The methodology developed is based on previous strength tests of the JA2 and SC propellants performed at different temperatures and strain rates [3-6], as well as on an elastomer compression standard [7].

2. PROJECTILE PROPELLANTS AS TEST MATERIALS

Modern gun propellants used today are so-called smokeless propellants. A smokeless propellant produces exclusively or predominantly gaseous products during combustion [8]. In view of their chemical composition, gun propellants can be classified as single-, double- or multi-base propellants.

2.1. Single-base propellants

Single-base propellants, also known as nitrocellulose (NC) propellants, are manufacturing by pressing a powder cake produced by gelling nitrocellulose with suitable solvents. The resulting powder cake mouldings undergo a drying process to strip the solvents [9]. The propellant grain obtained is porous.

2.2. Double-base propellants

Double-base propellants are fabricating by combining NC with nitroglycerine or dinitrodiglycol. The mixture thus obtained is centrifuged and the resulting mass is compacted by rolling. The thin sheet of powder cake obtained by rolling is called ‘powder cloth’. The powder cloth can then be cut into pieces called ‘powder flakes’. To obtain a propellant in the shape of tubes or rings, the powder cake is extruded through a suitable die and cut into tubes or discs (Fig. 1).

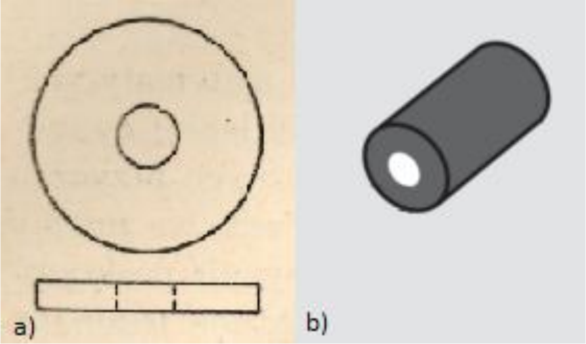

Fig. 1. Examples of propellant grain shapes: (a) Ring propellant [9], (b) Tube propellant [10]

The JA-2 propellant used in this study is an example of a two-base propellant. It consists of nitrocellulose, nitroglycerine and diethylene glycol dinitrate. Propellant grains are formed during the pressing process and can be regarded as a homogeneous rocket missile propellant.

Thanks to this manufacturing process, grains can be obtained without large deviations in roundness and with a high repeatability of geometrical dimensions [11]. The seven-channel JA - 2 propellant grain is shown at Fig. 2.

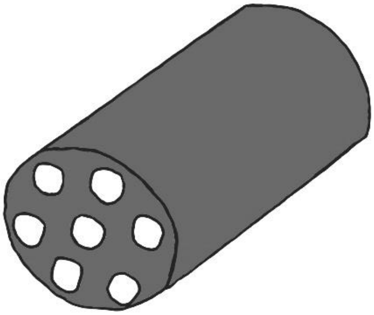

Fig. 2. Grain of the JA-2 propellant [10]

2.3. Multi-base propellants

Multi-base propellants are composites consisting of several phases with varying chemical or strength properties. An example of a multi-base propellant is SC propellant, which is an experimental gun propellant produced and tested at the Military University of Technology (MUT) in Warsaw (Poland). The main component of SC propellant is the high explosive material, i.e. hexogen (1,3,5-trinitro-1,3,5-triazene or RDX), and the exact composition of the gun propellant is proprietary information. The propellant grains have the same shape as in the JA-2 propellant (seven-channel Fig. 3).

Fig. 3. SC propellant grain [12]

Due to the laboratory method of production, the propellant grains are significantly deformed after the drying process (with a loss of cylindricity/parallelism of the end planes).

3. TEST METHODOLOGY

The test methodology is generally based on Polish standard PN-EN ISO 604:2006. This standard deals with the determination of the mechanical properties of plastics compressed at room temperature. Among other things, it defines the size and shape of the specimens and the crosshead speed, but does not standardise the parameters related to the cooling process itself. The parameters such as the method of specimen cooling, specimen temperature measurement and test temperatures were developed by the authors of this paper together with Michał Wójcik, MSc Eng. as part of his engineering thesis.

3.1. Specimen size and shape

The standard allows for cuboid, cylindrical or tubular specimens and the specimens may also have passageways (channels) [7]. The specimen dimensions are related to the expected maximum strain. The relationship between the dimensions and the maximum strain of the specimen is shown below (1).

$$\varepsilon_C^* \leq 0{,}4\frac{x^2}{l^2} \tag{1}$$

with:

ε_C^* – maximum nominal compressive strain during the test, expressed as a dimensionless ratio;

l – length of the specimen, measured parallel to the axis of the compressive force;

x – diameter of a cylinder, the external diameter of a tube, or the thickness of a cuboid measured relative to the shorter side of the cross-sectional area.

The maximum strain was assumed to be 50%. Hence, it follows from inequality (1) that $x^2/l^2 \geq 1{,}25$ and thus, $x/l \approx 1$. The only way to change the dimensions of the specimen is to reduce the length. Specimens of the SC propellant were also trimmed due to the lack of parallelism of the end planes of the propellant grains. The cross-sectional area of the SC propellant specimens, post-trimmed, was measured using a KEYENCE microscope (Fig. 4).

Fig. 4. Measurement of the cross-sectional area of specimens [12]

From the measured area, the theoretical diameter necessary to verify inequality (1) was determined. The diameter of the JA-2 propellant and the length of the specimens from both propellant grades were measured with a digital calliper. The examples of the dimensions of the SC propellant specimens are shown in Table 1.

Table 1. Dimensions of SC propellant specimens [12]

Surface area [mm^2]	**Theoretical diameter x [mm]**	**Specimen length l [mm]**	**Ratio of $\frac{x}{l}$**
26.902	5.85	4.75	1.23
26.626	5.82	4.93	1.18
25.584	5.70	4.65	1.23
24.061	5.53	4.63	1.19
25.731	5.72	4.54	1.26
25.509	5.70	4.75	1.20

All specimens were trimmed using a modelling hand saw and measured according to the methodology above-described.

3.2. Preparing the testing machine

Testing at sub-zero temperatures requires special instrumentation on the testing machine, i.e. a climate chamber. The MTS Criterion Model 45 testing machine equipped with a climate chamber was used (Fig. 5).

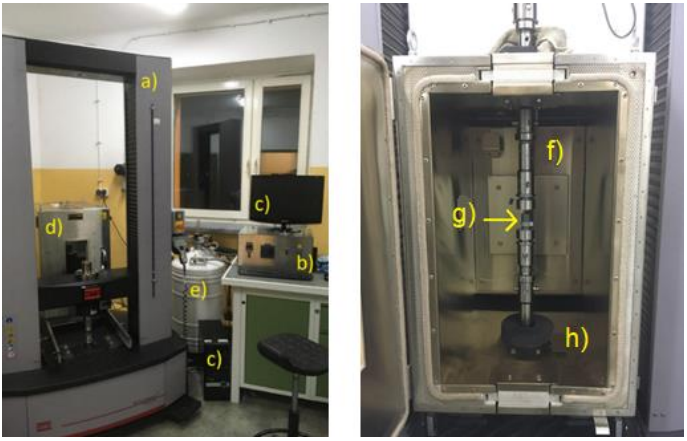

Fig. 5. Test stand: (a) testing machine, (b) climate chamber controller, (c) PC, (d) climate chamber, (e) liquid nitrogen tank, (f) fixture extension, (g) carbide shims, (h) thermal insulation [12]

Cooling was achieved by feeding liquid nitrogen into the climate chamber. The lowest temperature that could be achieved in the chamber was -100°C. In addition, a special set of fixture extensions had to be fitted to enable the test machine crosshead to be positioned above the climate chamber.

3.3. Test conditions

In addition to the specimen dimensions and fixture selection, two important parameters were considered: the deformation rate at which the specimen would be compressed (the crosshead speed) and the choice of temperatures at which the tests would be performed. PN-EN ISO 604:2006 strictly defines the permitted speed of crosshead movement (

Table 2). For the verification of the test methodology, the highest available crosshead speed, marked in green in Table 2, was chosen.

Table 2. Standardised crosshead speed values [7]

Crosshead speed [mm/min]	Permitted tolerance [%]
1	±20
2	±20
5	±20
10	±20
20	±10

The selection of test temperatures was based on the climate conditions in which the propellant can be used. They were not standardised as they were selected by the authors. The selected temperatures included room temperature (approximately 20-25°C) as the reference temperature and 0, -25, and -50°C. The lowest of these is the extreme temperature at which the propellant was expected to lose its desired strength properties and fracture completely.

4. VERIFICATION OF THE TEST METHODOLOGY

In order to verify the test methodology developed, at least three tests were performed on the propellant grades tested and for each temperature. In addition, the strain rate was determined for each specimen.

4.1. JA-2 propellant

Figure 6 shows the compression curves for different temperature conditions. In order to verify that the tests performed were reproducible, the curves plotted for the same temperature were collated on a single graph (Fig. 6).

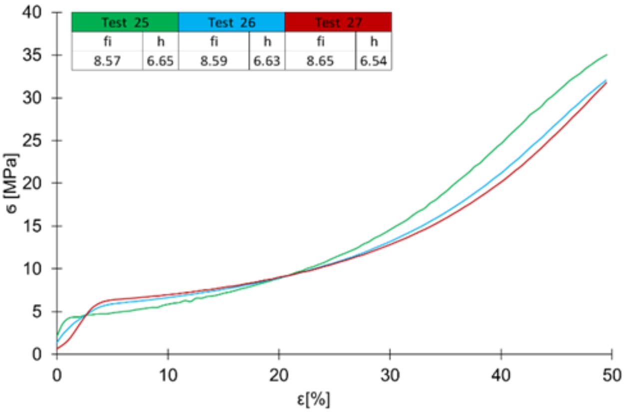

Fig. 6. JA-2 compressive stress-strain curves at 22 °C

Analysing the results obtained, it can be stated that the curves plotted were similar in profile, but differed slightly in the level of stress at large strain values. The JA-2 propellant deformed easily, i.e. plastic deformation occurred at a stress of about only 5 MPa. The tested specimens did not show any cracking or chipping. From the curves plotted, one (Test 26) was selected for further comparison with results obtained for other temperatures. A comparative chart is shown at Fig. 7.

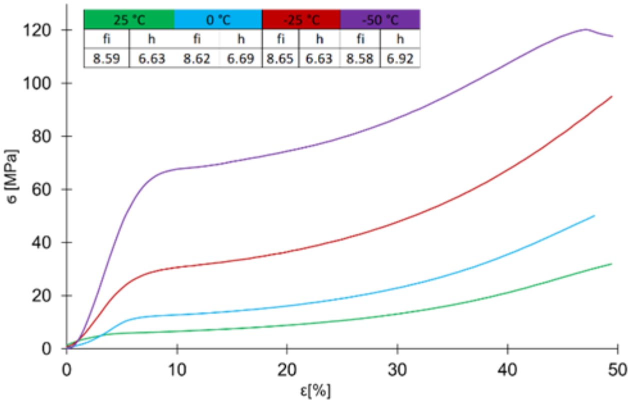

Fig. 7. JA-2 compressive stress-strain curves at different temperatures.

The change in temperature clearly affected the mechanical properties of the JA-2 propellant. The yield point of the material increased markedly. At room temperature, the propellant deformed plastically at a stress of about 5 MPa. This value increased as the temperature decreased:

- at 0ºC it was approximately 10 MPa;
- at -25ºC it was approximately 30 MPa;
- at -50ºC it was approximately 67 MPa.

The average strain rate for this propellant grade was $3.88 \cdot 10^{-2}$ s^{-1}.

4.2. SC propellant

Identical tests were carried out for the SC propellant grade. Due to the expected high brittleness of the propellant, some of the tests were completed at a lower strain than previously anticipated.

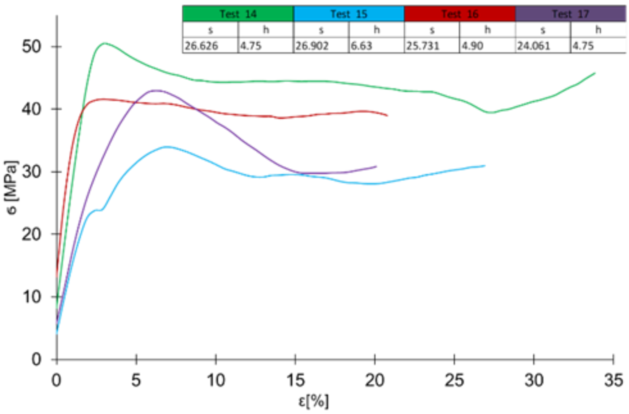

Fig. 8. SC compressive stress-strain curves at 22 °C

The SC propellant is much more brittle than JA-2. This is evident by the higher yield strength and the shape of the plot, suggesting a change in the cross-sectional area of the specimen during compression due to, for example, fracture. Despite four attempts at the tests, there was no clear similarity between compressive curves. Test No.16 was selected for further comparative analysis (Fig. 8). A summary of the results for all temperatures is shown in Fig. 9. They indicate that the SC propellant become increasingly brittle as the temperature decreased, and at sub-zero temperatures the propellant fragmented at small strain. The average strain rate obtained for SC propellant grade was $6.29 \cdot 10^{-2}$ s^{-1}.

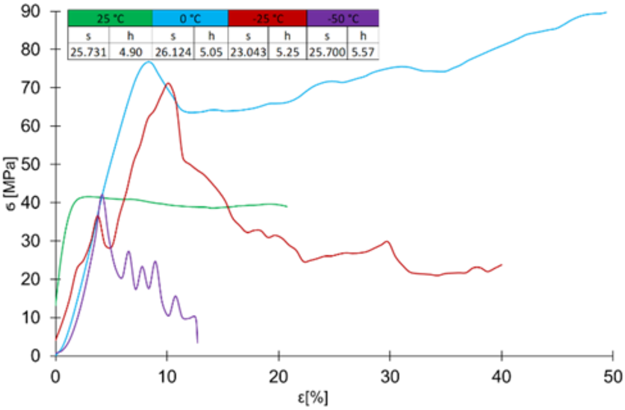

Fig. 9. Compression curves for SC at different temperatures

5. CONCLUSIONS

The obtained results confirmed that the experimental conditions used, i.e. the size of specimens and the method of test preparation and performance made it possible to obtain comparable and reliable results. This can be seen by analysing the results for the JA-2 propellant, which provide a high repeatability. The results obtained for this propellant grade are consistent with the data presented in [2-6]. Comparing the test results obtained with the references, it can also be seen that JA-2 propellant is sensitive to strain rate. In contrast, the strength tests on the SC propellant prove the validity of strength tests on this type of material. Brittleness of SC propellant grains is a very undesirable phenomenon. The results obtained are only a verification of the developed test methodology for high-energy materials at low temperatures. The strength tests should be repeated for different crosshead speeds. The test methodology developed can also be applied to other high-energy materials.

In the next stage of research work, the authors intend to test a selected grade of single-base propellant, solid rocket propellants (for ejected aircraft seats, for example), and low-sensitivity explosives, like TNT or hexogen. In addition, the materials will be tested for phase transitions during temperature decreasing.

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Metodyka badania materiałów wysokoenergetycznych w warunkach obniżonej temperatury

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Streszczenie. W artykule przedstawiono opracowaną metodykę badania prochów w obniżonej temperaturze na podstawie normy PN-EN ISO 604:2006. Opisano krótką charakterystykę badanych materiałów oraz najważniejsze warunki przeprowadzania statycznej próby ściskania takie, jak: wymiary próbek, prędkość deformacji oraz zakres temperatur dla wybranych gatunków prochu, tj.: JA-2 oraz SC. W celu weryfikacji opracowanej metodyki wykonano wstępne badania wytrzymałościowe w wybranych temperaturach (25, 0, -25 i -50 °C). Testy przeprowadzono dla próbek powstałych poprzez skrócenie ziarna do wymiarów zgodnych z normą. Uzyskane wyniki potwierdziły oczekiwane właściwości wytrzymałościowe dla obu gatunków prochu (kruchość, wytrzymałość na rozciąganie). Proch JA-2 ze względu na skład chemiczny jest materiałem o niskiej kruchości nawet w temperaturze -50 °C. Nie ulega całkowitemu skruszeniu, rośnie jedynie jego granica plastyczności. Wyniki uzyskane dla prochu JA-2 są zbieżne z wynikami opublikowanymi w literaturze. Proch SC okazał się materiałem bardzo kruchym już w temperaturze pokojowej. W temperaturach poniżej 0 °C ulegał całkowitemu skruszeniu po osiągnięciu zakładanego odkształcenia. Uzyskane wyniki potwierdzają, że przyjęte warunki testu wytrzymałościowego oraz sposób przygotowania i przeprowadzenia badań umożliwia uzyskanie porównywalnych i wiarygodnych wyników. Można to zauważyć analizując wyniki dla prochu JA-2, który są zgodne z danymi zamieszczonymi w dostępnej literaturze. Badania prochu SC udowadniają natomiast zasadność badań wytrzymałościowych tego typu materiałów. Kruchość ziaren prochowych jest zjawiskiem bardzo niepożądanym. Zmiana powierzchni spalania prochu wynikająca z procesu pękania ziaren prochowych może wpłynąć negatywnie na wielkość i przebieg impulsu ciśnienia i doprowadzić do zniszczenia komory nabojowej lub lufy działa.

Słowa kluczowe: inżynieria mechaniczna, badania w obniżonej temperaturze, badania wytrzymałościowe, statyczna próba ściskana, prochy artyleryjskie