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On Standards for Mechanical Testing of Materials at Cryogenic Temperatures

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О стандартах на механические испытания материалов при криогенных температурах

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Рассмотрены и проанализированы основные положения стандартов ASTM, ISO и ГОСТ, которые регламентируют метод испытаний металлов на растяжение при температуре жидкого гелия. Определены основные факторы, обусловливающие параметры деформирования, нагружения и размеры образцов при испытаниях металлов и сплавов в указанных температурных условиях. Представлены рекомендации по дополнению и изменению положений стандартов.

Ключевые слова: нормативный документ, прерывистое течение, податливость машины, энергоемкость материала, температура жидкого гелия, скорость деформирования, скорость нагружения.

Introduction. Investigations on mechanical properties of structural metallic materials at temperatures close to absolute zero, which were developed extensively in the 60th–80th of the last century, have not lost their importance up to now as evidenced by numerous publications of both Ukrainian and foreign researchers [1–7]. This interest in the problem, which would, as it might seem, concern only experts specializing in particular, quite narrow, and even specific fields of human activity (space exploration, defense industry), is connected with the fact that cryogenic temperatures have already been used since some time and will be introduced even faster in certain spheres of our day-to-day life (power engineering, medicine, agriculture, electronic engineering). The range of devices whose units should be in operation at very low temperatures is expanding accordingly. The carrying capacity of these units is ensured by metallic materials. The immediate interest in such investigations is emphasized by the fact that ASTM E 1450 standard (2003) and ISO 19819 standard (2004), which regulate the methods for testing metallic materials in liquid helium, have been recently developed in the USA and the European Union. The State Standard of the USSR GOST 22706-77 “Metals. Methods for Tensile Testing at Temperatures Ranging from −100 to −269°C” was developed in the USSR in 1977 and brought into effect in 1978 in order to unify the methods for determining mechanical characteristics of metallic materials at cryogenic temperatures. This normative document as amended has been effective in Ukraine up to now. Essentially GOST 22706-77 is a version of the State Standard GOST 1497-73 “Metals. Methods for Tensile Testing” (next wording of which STSEV 471-77 appeared in 1984) that was extended to include the procedure of cooling down to temperatures of 77 and 4.2 K. Its original version regulates testing at a temperature of 293 K and practically does not consider the peculiarities of the development of the elastic-plastic deformation of metallic materials at the temperature of liquid helium. These peculiarities, namely, the unstable accumulation of plastic strain by metals and alloys loaded under the above temperature conditions (the so-called discontinuous yielding, see Figs. 1 and 2), determine basic requirements for the parameters and modes of tensile testing of metallic materials at a temperature of 4.2 K.

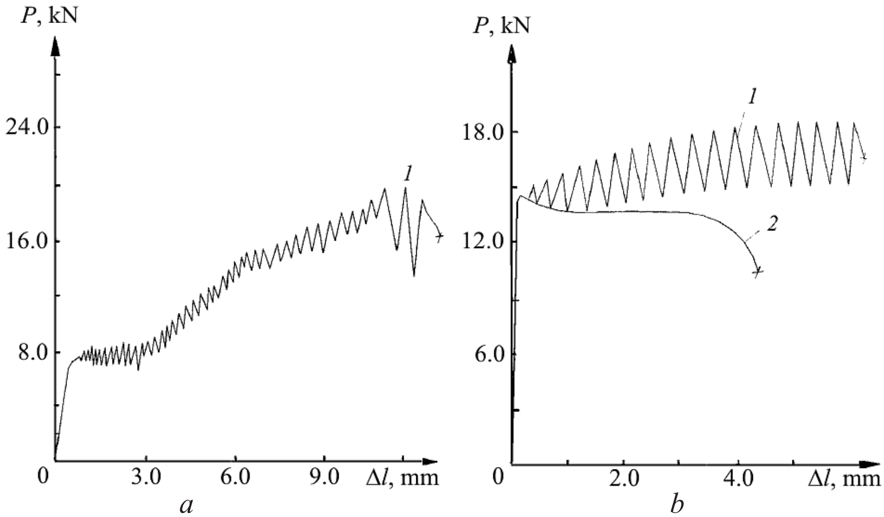

Fig. 1. Load–elongation diagrams for steels 12Kh18N10T (*a*) and 03Kh20N16AG6 (*b*) at a temperature of 4.2 K: (*1*) constant strain rate; (*2*) constant loading rate (within the elastic region); specimen $\varnothing_0 = 4$ mm, $l_0 = 20$ mm.

Analysis of the main factors set forth or omitted in the clauses of the ASTM E 1450:03, ISO 19819:04 and GOST 22706-77 standards, which determine the kinetics of discontinuous yielding in materials and alloys and, as a consequence, the values of their mechanical characteristics, will be presented hereafter.

Discussion. Table [7] gives the values of the mechanical characteristics of steels and aluminum alloys obtained under similar temperature conditions (in liquid helium) but on specimens of different geometry and dimensions, under different loading conditions (constant cross-head speed or constant loading rate), and at different ratios of the energy absorbed by the specimen and the elastic energy accumulated by the loading system of the testing facility (hereinafter referred to as the machine).

As we can see, the extent to which the above factors influence the values of the characteristics is ambiguous. By the extent of this effect the above factors can be placed in the following order: specimen shape, specimen dimensions; loading rate and conditions; the relation between the energy absorbed by the specimen and the amount of the elastic energy accumulated in the machine. The shape of the specimen cross-section (either circular or rectangular) under conditions of discontinuous yielding must ensure maximum approximation to the linear stress state. Dimensions of the specimens determine the requirements for the systems of loading, cooling, mechanical force exciter, etc., on the one hand, and, on the other hand, regulate the development of elastic-plastic deformation, i.e., the number and amplitude of the load jumps and the energy absorbed by a specimen. According to references [3, 8], the strain rate below $7 \cdot 10^{-4}\ s^{-1}$ has no effect on the parameters of discontinuous yielding in metals and alloys. At the same time, the effect of the strain rate on the discontinuous yielding alone has been considered, i.e., the number of jumps, their amplitude and shape. There are no systematic data on how the mechanical characteristics of metals and commercial alloys change in response to changing the strain rate.

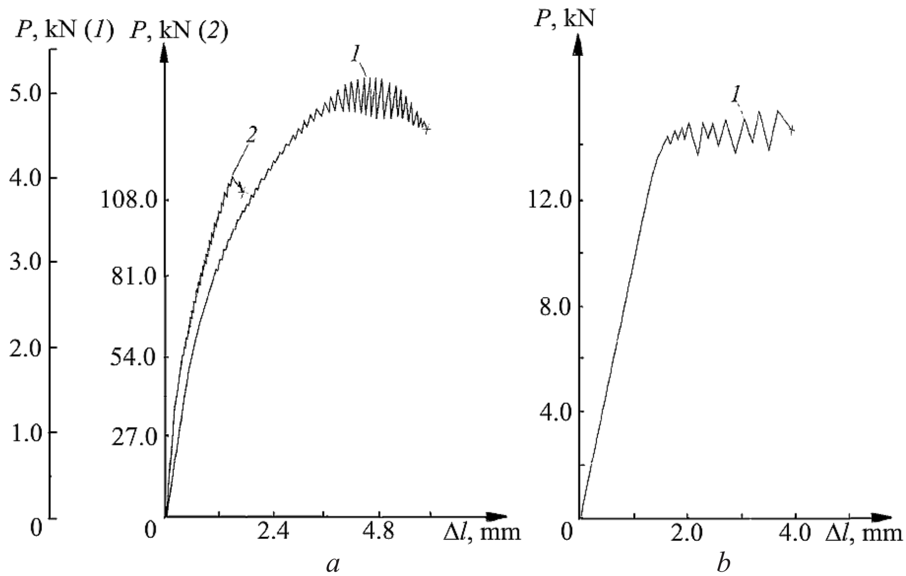

Fig. 2. Load–elongation diagrams for aluminum alloy AMg6 (a) and titanium alloy 19 (b) at a temperature of 4.2 K and a constant strain rate: (*1*) specimen $\varnothing_0 = 4$ mm, $l_0 = 20$ mm; (*2*) specimen 12×20 mm, $l_0 = 80$ mm.

Nevertheless, since the discontinuous yielding kinetics also determines the values of the material strength and plasticity characteristics, it can be stated that at a strain rate of $\dot{\varepsilon} \leq 10^{-3}\ s^{-1}$, as put forth in ASTM 1450 and ISO 19819 standards, the values of the mechanical characteristics will be independent of the strain rate as well. All the standards analyzed in this paper regulate the strain rate alone (cross-head displacement). This is specifically emphasized in the ASTM standard. Nevertheless, at a sufficient rigidity of testing facilities the values of the mechanical characteristics obtained at a constant loading rate agree closely with analogous values obtained at a constant strain rate [9]. That is, the standards must also set the regimes of specimen loading at a constant rate of load application for a given rigidity (compliance) of a testing machine.

By far and large, the development of discontinuous yielding depends on the elastic energies accumulated in the force excitation and load transmission systems of the testing machine during the specimen loading, or, more specifically, on the ratio of the specimen energy capacity to the total elastic energy accumulated in the force excitation and load transmission systems, and on the rate of this elastic energy reproduction. For instance, in steel 03Kh20N16AG6 with a stable austenite structure, the number of the load jumps decreases from 22 at a constant strain rate (insufficient amount of the machine elastic energy) to 1 at an uncontrolled increase in the loading rate (considerable amount of the machine elastic energy and high speed of its reproduction) during the jump. Accordingly, the determined ultimate strength of the steel decreases 1.20 times, relative elongation after fracture 1.34 times, and relative necking after fracture due to strain localization increases 1.40 times.

The elastic energy W_m accumulated by the force excitation and the load transmission systems of the testing machine during the specimen loading can be presented in the general form as follows:

$$W_m = \sum_{i=1}^{n} W_i (L_i, F_i, E_i, P) + W_f (P), \tag{1}$$

where W_i is the elastic energy accumulated by the ith element in the load transmission system during its loading to the load level P, E_i is the material elastic modulus, L_i is the length, F_i is the area of the element cross section, n is the number of elements in the force excitation system, and W_f is the elastic energy of the force excitation system accumulated during loading to the P level.

The capacity of an element to accumulate elastic energy is characterized by its compliance, C_i, i.e., the ratio of the element absolute elastic elongation Δ_i^e to the load value P that induces this elongation:

$$C_i = \frac{\Delta_i^e}{P}. \tag{2}$$

The higher the system compliance, the higher the amount of the elastic energy accumulated in the system at equal loads. The element energy during tension is

$$W_i = \frac{P \Delta_i^e}{2} = C_i \frac{P^2}{2}. \tag{3}$$

The influence of the amount of the accumulated elastic energy shows itself mainly after a decrease in the material resistance to deformation, or, as stated by the authors of [10], at the supercritical stage. Discontinuous yielding of metallic materials at a temperature of 4.2 K is formed by a number of consecutive events of local deformation of some parts and the gauge length of a specimen, each bringing about local reduction in the cross section area – necking. Throughout this process, the elastic energy accumulated by the machine, Eq. (1), and in the specimen region beyond the neck is released. This energy maintains local deformation. That is, every event of discontinuous yielding can be considered as the material transition to the supercritical stage. The development of the local deformation at the supercritical stage can discontinue only if the residual elastic energy of the machine–specimen system (after one event of discontinuous yielding) corresponds to the residual energy capacity of the specimen material:

$$W'_{res} = W'_m + W_{nds}, \qquad (4)$$

where W'_{res} is the residual energy capacity of the specimen in a local zone, W'_m is the residual elastic energy of the machine, and W_{nds} is the residual elastic energy of the plastically non-deformed part of the specimen.

The residual energy capacity of the specimen material decreases after every event of discontinuous yielding. That is why the smaller the total compliance of the elements of the load transmission and force excitation systems, the smaller the probability of premature specimen fracture after the next strain jump. At $W_m \to 0$ ($C_m \to 0$) it is possible to determine the total deformability of the material of specimens of certain dimensions. In testing specimens with the same dimensions on a machine with a greater compliance, fracture will occur earlier. The extent of premature fracture depends on the specimen dimensions, material properties (energy capacity), compliance of the load transmission and force excitation systems, and the rate of supplying mechanical energy to the specimen. As emphasized above, the development of discontinuous yielding determines the values of the metallic material strength and plasticity characteristics, therefore, when setting the requirements for testing equipment, it is necessary to consider not only the strain rate, as it was done in the documents considered in this paper, but also the machine compliance, Eq. (4). In doing so it is also necessary to take into account the fact that standard machines used for static tensile tests of metals at a temperature of 4.2 K have three types of the load excitation systems: mechanical, hydraulic, and servohydraulic ones. Machines equipped with a mechanical load excitation system ensure deformation mostly at a constant rate, those with hydraulic ones – at a constant loading rate (usually the constancy is provided within the zone of the specimen elastic deformation), and those with servohydraulic load excitation system realize both regimes of load application (yet modern machines of this type with a small compliance ensure the constancy of the loading rate practically up to the moment of the specimen fracture). The main accumulators of the elastic energy in the machines of the first and the third types are the elements of the machine load transmission system. The energy accumulated by the force excitation system is insignificant and, as a rule, $W_f << \sum_{i=1}^{n} W_i$. In hydraulic machines, especially of a direct action, this value is significant and can be far above $\sum_{i=1}^{n} W_i$. This proceeds from the following considerations.

The value of the elastic energy accumulated by a hydraulic force excitation system is proportionate to the condensability of the liquid β used as a working medium of the force excitation system. The condensability β is a reciprocal of the bulk modulus of elasticity of the material K. For steel at a temperature of 293 K the bulk modulus of elasticity is

$$K = \frac{1}{\beta} = \frac{E}{3(1-\mu)} = 1.52 \cdot 10^6 \text{ kg/cm}^2.$$

Here E and μ is the elasticity modulus and Poisson's ratio, respectively. That is $\beta = 0.66 \cdot 10^{-6}$ atm^{-1}.

The condensability of oil used in the force excitation systems of hydraulic machines with the pressure scale from 1 to 10 atm is within the range of $(47.2\text{–}63.3) \cdot 10^{-6}$ atm^{-1}. Thus, the oil compliance is from 60 to 100 times greater than that of steel. The elastic energy accumulated accordingly by a hydraulic load excitation system will be ~ (60–100) times higher as compared to metallic elements of the load transmission system, all other conditions being equal (temperature, material volume, etc.). Availability of such considerable amounts of the elastic energy accumulated in the machine systems does not mean that they cannot be used for tensile testing of metals and alloys at a temperature of 4.2 K. It is generally known that some structures, for instance, pressure vessels, operate under non-steady-state loading. In order to carry out strength calculations for such structures operated at temperatures close to 4.2 K, to evaluate their load carrying capacity, life, etc., the mechanical characteristics of materials should be determined when the testing machine systems have accumulated certain amounts of the elastic energy.

Thus, standards should regulate not only the strain rates or loads (which none of those being analyzed does) but also the testing machine compliance.

Specimens. In tests in the medium of liquid helium, the specimen size and geometry determine not only the design peculiarities of the cooling systems, cryostat, grips, the maximum load capacity of the testing machine but also the kinetics of the material deformation thus defining the strain rate, the rigidity of the stress state in the necks, and the ratio of the elastic energies accumulated in the machine–specimen system, i.e., the values of the material mechanical characteristics that are determined in the course of testing (see Table 1).

As seen from the data presented here, the number of events of discontinuous yielding and their amplitude and, as a consequence, the values of the material strength and plasticity characteristics also depend considerably on the size and shape of the specimen, even if it meets the requirements of the State Standard GOST 22706-77. Therefore, it is reasonable that the dimensions of specimens for tests at the temperatures of liquid helium should be limited (in contrast to GOST 22706-77) by the requirements of normative documents. To obtain reliable values of the mechanical characteristics and for their comparability, the normative documents should regulate one basic standard size of a cylindrical specimen. This can be a small standard specimen recommended by ASTM E 1450:03. For sheet materials of small thickness, from which the manufacture of this specimen by orienting it across the direction of rolling is impossible, it is advisable to use a plane specimen described in ISO 19819. It is the values of the mechanical

T a b l e 1

Mechanical Properties of Steels and Alloys at a Temperature of 4.2 K

Material	$S_0, \varnothing_0$, mm	$\sigma_{0.2}$, MPa	σ_u, MPa	δ, %	ψ, %	n	J_c, kJ/m^2
AMg5	$S_0 = 12.0$ mm $\varnothing_0 = 4.0$ mm	192 170 (170)	465 540 (405)	11.0 41.5 (32.0)	10.0 28.5 (40.0)	235 93	17.0*
AMg6	$S_0 = 12.5$ mm $\varnothing_0 = 4.0$ mm	197 135 (135)	517 547 (470)	18.5 24.0 (22.5)	14.5 15.5 (27.2)	203 74	5.0*
AMtsS	$S_0 = 10.0$ mm $\varnothing_0 = 4.0$ mm	150 147 (140)	400 425 (340)	32.0 29.0 (28.0)	33.0 27.0 (30.0)	272 103	44.5
03Kh20N16AG6	$S_0 = 9.0$ mm $\varnothing_0 = 4.0$ mm	1150 1290 (1293)	1620 1640 (1305)	34.0 35.5 (22.5)	40.0 36.0 (51.0)	34 22 (1)	150.0*
0N9	$S_0 = 10.0$ mm $\varnothing_0 = 4.0$ mm	1040 1230 (1240)	1481 1560 (1300)	19.5 20.0 (16.0)	48.0 51.5 (67.5)	21 14 (1)	18.0**
12Kh18N10T	$\varnothing_0 = 4.0$ mm	770 (775)	1680 (1415)	44.5 (39.0)	34.5 (60.5)	49 (11)	101.0

Notes. The magnitude of the characteristic at $W_m > W_{sp}$ is given in brackets; * correspond J_{Ic} (critical value of J-integral); ** correspond to the value of J_c calculated with the account taken of K_{Ic}; S_0 and $\varnothing_0$ are specimen thickness and diameter, respectively; $\sigma_{0.2}$ is offset yield stress; σ_u is ultimate strength; δ is relative elongation after fracture; ψ is relative necking after fracture; n is the number of discontinuous yielding events.

characteristics obtained using these unified specimens that should be included in handbooks and it is such values that should be the object of comparison. In order to minimize the influence of the energy accumulated in the machine on the deformation kinetics of a specimen, its dimensions should be selected in such a way as to ensure a certain ratio between the energy absorbed by the specimen and the elastic energy accumulated by the machine:

$$\frac{W_{sp}}{W_m} = n, \qquad n > 1. \tag{5}$$

The greater the value of n, the smaller (when going to a supercritical phase) the effect of the elastic energy accumulated by the machine on the kinetics of further specimen deformation. If, with the use of unified specimens, requirement (5) is not met, the compliance of the machine elements should be adjusted.

In view of (3), the energy accumulated by the machine at the load P can be defined as

$$W_m = \sum_{i=1}^{n} W_i = \sum_{i=1}^{n} C_i \frac{P^2}{2}. \tag{6}$$

Here C_i is the compliance of the machine elements, i.e., the components of the load transmission system, their coupling, and the load excitation system.

Since after each event of discontinuous yielding the specimen is deformed in an elastic manner, in order to meet requirement (5), it is sufficient to fulfill the following condition to a first approximation:

$$C_m \le C_{sp}, \tag{7}$$

where $C_m = \sum_{i=1}^{n} C_i$.

If the gauge length of the specimen is a geometrically uniform rod, then

$$C_{sp} = \frac{\Delta_{sp}}{P} = \frac{l_0}{F_0 E}, \tag{8}$$

where l_0 and F_0 are the initial calculated gauge length and the cross-section area of the specimen, respectively, E is the modulus of elasticity of the material at the test temperature, and Δ_{sp} is the absolute elastic elongation of the specimen at the load P.

It is obvious that a change in the specimen dimensions results in a change in its compliance, i.e., the ratio between the elastic energies of the machine and the specimen can be violated, therefore, condition (7) should be written as

$$C_m \le \frac{l_0}{F_0 E}.$$

One must also consider that, in the case of discontinuous yielding, the value of l_0 corresponds to a part of the initial calculated specimen length l_0' that is deformed at every event of deformation. Using the approach presented in [11], it is possible to show that for a five-fold cylindrical specimen $l_0' \cong \frac{2}{15} l_0$. The remaining 13/15 of the initial calculated specimen length is the source of the elastic energy that contributes to the material yielding in a localized zone l_0'. That is, the compliance of the specimen is defined only by a part of its gauge length. It is in the volume of this part of the specimen during the event of discontinuous yielding that the elastic energy accumulated by the machine and the specimen outside this part is released. In the final form, condition (7) for cylindrical five-fold specimens can be written as

$$C_m \le \frac{2 l_0}{15 F_0 E}.$$

At the same time, normative documents should allow defining the mechanical characteristics of materials using specimens of other dimensions (according to ASTM E8 or GOST 1497) either at the amount of the elastic energy accumulated by the machine that differs from condition (7) or at other loading regimes if this is stipulated by the conditions of the material operation in structures or by the requirements to metal products. These statements should be specified by separate paragraphs of documentation.

Additional Remarks on the Standards. The strain rate in the interval indicated in the standard should not influence the values of the characteristics determined in accordance with the standardized procedure. Therefore, the statements of paragraphs 9.5.4 (ASTM 1450-03) and 8.3.3 (ISO 19819:2004) are subject to discussion. First, if the strain rate variation in the regulated range leads to a change in the deformation behavior or in the values of the mechanical characteristics, this means that the selected range of rates does not ensure the identity of the testing conditions. Second, the requirements of changing the strain rate for determining the offset yield stress if the first event of the discontinuous yielding takes place at the residual strain of less than 0.2%, which are stated in paragraphs 9.5.4 and 8.3.3 of the corresponding standards, are erroneous in essence. In our opinion, in this case, the yield strength should be determined from the stress, which corresponds to the onset of the first load jump. This manifestation of yielding is the property of the material, i.e., the stress at which this jump takes place is the yield strength under given test conditions, in particular, at a given strain rate.

In normative documents, it is reasonable to recommend that the compliance of the force excitation and load transmission systems of the testing machine should be determined experimentally. For this purpose it is necessary to use the method of a low-compliance specimen [12]. Incidentally, the data on the machine compliance obtained using this method make it possible to adjust the slope angle of the elastic line of the tensile curves in the case of measuring the specimen elongation not directly on its gauge length. This approach should also be included in the recommendations of normative documents since measuring the specimen elongation directly on the gauge length in the medium of liquid helium is a rather difficult problem and when the amount of investigations is large it difficult to perform.

Conclusions. The normative documents that regulate the methods for testing metallic materials at a temperature of liquid helium (ASTM E 1450-03, ISO 19819, edition of 2004, GOST-22706-77) have common essential drawbacks, which have to be eliminated when developing an appropriate National Standard of Ukraine. Among other things it is necessary:

(i) to regulate the compliance of the testing machine (the ratio of the machine–specimen compliances);

(ii) to make it mandatory to test specimens of a unified standard size;

(iii) to determine the yield strength from the stress at which the first event of discontinuous yielding begins or the offset yield stress from the stress that corresponds to a normalized level of residual strain (the generally accepted level is 0.2%) for the selected standard strain rate.

Р е з ю м е

Розглянуто і проаналізовано основні положення стандартів ASTM, ISO та ГОСТ, що регламентують метод випробувань матеріалів на розтяг при температурі рідкого гелію. Виявлено головні фактори, що обумовлюють параметри деформування, навантажування та розміри зразків при випробуваннях

металів і сплавів в умовах указаної температури. Подано рекомендації щодо доповнень положень стандартів.

1. T. J. Burns, "A simple criterion for the onset of discontinuous deformations in metals at very low temperatures," *J. Mech. Phys. Solids*, **42**, No. 5, 897–811 (1994).
2. M. M. Krishtal, "Frequency sensitivity of the resistance to deformation and strain macrorealization of during discontinuous yielding of Al–Mg alloys," *Metalloved. Term. Obrab. Metal*., No. 9, 26–30 (1997).
3. V. V. Pustovalov, "The effect of superconducting transition on the low-temperature jump-like deformation of metals and alloys (Review)," *Fiz. Nizk. Temper*., No. 6, 515–535 (2000).
4. A. K. Emaletdinov, "Unstable modes of plastic deformation of metals at helium temperatures," *Fiz. Metal. Metalloved*., **91**, No. 4, 3–9 (2001).
5. S. A. Vologzhanina, Yu. P. Solntsev, T. V. Ermakova, "The effect of structural changes on the reliability and service life of the material for cryogenic equipment," in: *Strength of Materials and Structures at Low Temperatures* [in Russian], Collected Papers of the St. Petersburg State University, St. Petersburg (2002), pp. 7–25.
6. E. V. Vorob'ev and T. V. Anpilogova, "Peculiarities of manifestation of the effect of low-temperature jump-like deformation," in: *Reliability and Life of Machines and Structures* [in Russian], Kiev (2006), Issue 26, pp. 166–174.
7. L. S. Novogrudskii, "Evaluation of low-temperature hardening of structural materials at a temperature of 4.2 K," in: *Reliability and Life of Machines and Structures* [in Russian], Kiev (2006), Issue 26, pp. 319–325.
8. V. I. Startsev, V. Ya. Illichev, and V. V. Pustovalov, *Plasticity and Strength of Metals and Alloys at Low Temperatures* [in Russian], Metallurgiya, Moscow (1976).
9. V. A. Strizhalo, N. V. Filin, B. A. Kuranov, et al., *Strength of Materials and Structures at Cryogenic Temperatures* [in Russian], Naukova Dumka, Kiev (1988).
10. Ya. B. Fridman, T. K. Zilova, B. A. Drozdovskii, *Kinetics of Deformation and Fracture* [in Russian], VHNIIAM, Moscow (1960).
11. V. I. Eremin, "Geometry of the zone of localized deformation in low-temperature discontinuous yielding of metals," *Strength Mater*., **19**, No. 2, 181–184 (1987).
12. V. O. Stryzhalo, L. S. Novogrudskyi, M. P. Zemtsov, *Method to Determine the Energy- and Strain-Based Characteristics of a Structural Material* [in Ukrainian], Ukraine Inventor's Certificate 69351A, MKI G01N3/00 No. 20031212975. Bull. No. 8 (2004).

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