Application of the Traceability Concept in Determining the Mechanical Properties of Metals Under Static Tension Using a GSO 11854-2021 Reference Material

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Abstract This paper deals with the specific features of applying approved reference materials as a tool for ensuring traceability and controlling the accuracy of mechanical property measurements. The authors analyzed the theoretical approach based on ISO/IEC Guide 98-3:2008 and calculation algorithms provided by ISO 21748:2017 for evaluating measurement uncertainty. The methodology of using a GSO 11854-2021 certified reference material of St20 steel mechanical properties for evaluating the uncertainty of static tensile test measurements is considered. In order to ensure the traceability of measurement results, two options for accounting the laboratory systematic component are proposed: as a correction or a contribution to the standard combined uncertainty. According to the conducted study, the modeling approach of theoretical concepts based on ISO/IEC Guide 98-3:2008 and calculation algorithms provided by ISO 21748:2017 (Eq. 1) can be applied by accredited laboratories when assessing uncertainty in accordance with clause 7.6 of GOST ISO/IEC 17025-2019.

Keywords Mechanical properties · Static tension · Metrological traceability · Primary reference measurement technique · Reference material

Introduction

Determination of the mechanical characteristics of metals under static tension is a key method for identifying their strength and ductility indicators. Static tensile tests represent indirect measurements, where strength (tensile strength, proof strength) and ductility (percentage elongation after fracture, percentage total extension at fracture) are determined using corresponding measurement equations under specified test conditions. In this regard, although the results of determining mechanical properties cannot be correlated with a comparison base in the form of a physical quantity

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reference, either a primary reference measurement procedure or a reference material (RM) can be applied. However, the use of RMs or primary reference measurement procedures as a comparison base is associated with a number of specific features, which are not immanent to quantitative chemical analysis. These features arise due to the heterogeneity of either RMs or the material used for comparing the results of a laboratory procedure according to GOST 1497-84 [1] with those obtained by a primary reference measurement procedure.

Bahng and Roebben [2–4] classify mechanical properties as characteristics that depend on test procedures. A confusion in establishing the traceability of mechanical properties is caused by expressing the results of testing the mechanical properties of metals in SI basic units. Thus, strength in static tensile tests is measured in MPa, which are the units of pressure (force, divided by the area). However, the assumption that the properties of metals in static tensile tests can be traced to force 01basic units, seems to be incorrect, since these properties are measured prior to sample destruction. In order to obtain a response from a sample, it is necessary to use an external action in the form of tension, e.g., under a certain deformation rate. This means that any variations in the method or procedure of external action will affect the measurement results. Therefore, an RM is needed to ensure the traceability of properties and to control the accuracy of applying a particular test method.

Adamczak et al. [5–7] discussed various approaches to calculating the uncertainty of strength and ductility characteristics. An essential drawback of these studies involves uncertainty budgeting only on the basis of data on the traceability to the units of force and length, i.e., without using an RM. Tolmachev and Matveeva [8] considered an approach to ensuring the metrological traceability of the results of measuring mechanical properties during static tensile tests to a primary reference measurement technique using RMs. When evaluating the uncertainty of static tensile test results, a contribution of traceability, assessed using RMs, requires accounting for the laboratory systematic component either as a correction or as a contribution to the standard combined uncertainty.

In this paper, we aim to study and verify a methodology of using RMs to establish traceability in determining the mechanical properties of metals under static tension. To this end, the following research objectives were formulated: to analyze the theoretical principles specified in ISO/IEC Guide 98-3:2008 [9] and calculation algorithms proposed by ISO 21748:2017 [10] for evaluating measurement uncertainty; to evaluate the uncertainty of static tensile test results using the example of a GSO 11854-2021[1] reference material of the mechanical properties of St20 steel; to develop an approach based on uncertainty budgeting taking into account the combined effect from all sources of uncertainty, including bias, caused by traceability.

[1] GSO 11854-2021 Reference materials of the approved type of mechanical properties of steel grade 20. Available via FIF EUM. https://fgis.gost.ru/fundmetrology/registry/19/items/1395637. Accessed 15 October 2022 (In Russ.).

Table 1 Metrological characteristics of GSO 11854-2021 CRM of the mechanical properties of grade 20 steel

Certified characteristic	RM certified value	Absolute expanded uncertainty of certified values ($P = 0.95$, $k = 2$)
Tensile strength R_m, MPa (N/mm^2)*	446	± 6
Lower yield strength R_{eL}, MPa (N/mm^2)*	250	± 10

* For proportional cylindrical samples according to GOST 1497-84 with the original gauge length $l_0 = 5d_0$, where d_0 is the original diameter of the parallel length of a circular test piece

Materials and Methods

According to JCGM 200:2012 [11] (clause 2.41), metrological traceability is the property of a measurement result whereby the result can be correlated with a reference system through a documented continuous chain of calibrations, each contributing to the measurement uncertainty. In this research, we study the mandatory characteristic of the result of measuring the strength properties of a metal, i.e., the uncertainty of tensile strength, and carry out an analysis of traceability contribution to uncertainty budgeting. The GSO 11854-2021 certified reference material (CRM) of the mechanical properties of St20 steel was selected as a comparison base.

A GSO 11854-2021 approved type CRM was obtained as a result of the study and characterization of steel hot-rolled circular products according to GOST 2590-2006 [12] made of St20 steel according to GOST 1050-2013 [13]. The characterization of rolled samples in terms of their tensile strength and lower yield strength was carried out on the State standard of the unit of force of the first category[2] provided by GOST 1497-84. The standard uncertainty of the characterization was 0.9 N/mm^2. In order to evaluate the expanded uncertainty of certified values, during the type approval tests, the standard uncertainty from the heterogeneity of the reference material equal to 2 N/mm^2 was established.

Table 1 provides the metrological characteristics of the CRM of the mechanical properties of St20 steel, established as a result of testing batch No. 1.

Various approaches can be used to evaluate measurement uncertainties. All of them include determination of the measurand and careful identification of all possible contributions to an increase in the uncertainty of measurements.

Figure 1 illustrates the classification of approaches to the evaluation of uncertainty proposed in [14]. The classification is based on the difference between the evaluation of uncertainty, conducted by the laboratory itself (intralaboratory approach) and uncertainty, based on combined studies in various laboratories (interlaboratory approach).

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[2] GET 32-2011 State working standard of the unit of force of the first category in the range of values from 1 to 50 kN. Available via FIF EUM. https://fgis.gost.ru/fundmetrology/registry/11/items/415290. Accessed 15 October 2022 (In Russ.).

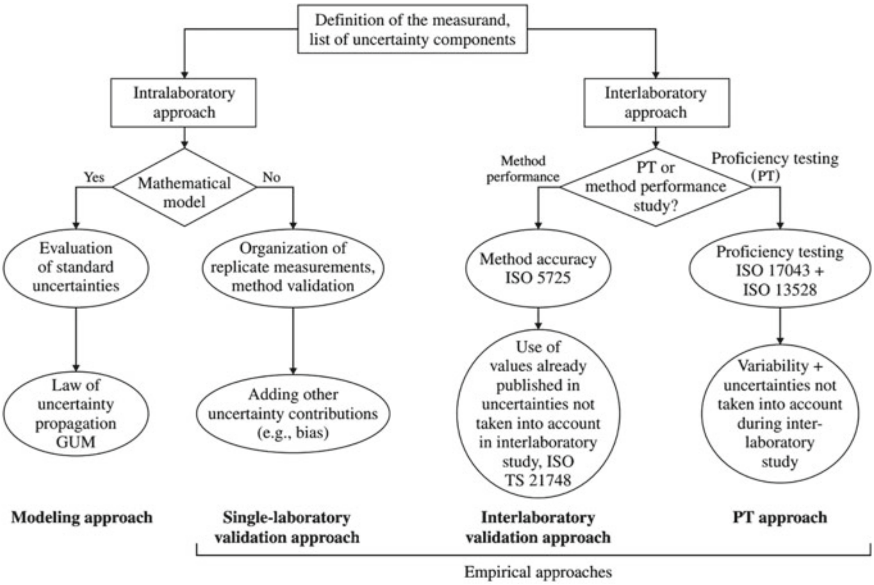

Fig. 1 Classification of approaches to the evaluation of uncertainty according to [9]

In this study, we used a modeling approach based on ISO/IEC Guide 98-3:2008. A mathematical model was created, representing an equation that determines the quantitative relationship between the measurand and all dependent quantities, including all components that contribute to the uncertainty of measurements. An evaluation of the standard uncertainties of all individual components of uncertainty was made. Standard deviations of repeated measurements are directly the standard uncertainties for the corresponding components (given the normal distribution). The combined standard uncertainty is calculated by applying the uncertainty propagation law, which depends on the partial derivatives for each input value. The expanded uncertainty U (providing the interval from $(y - U)$ to $(y + U)$ for the measurand y is calculated. For a normal distribution, the coverage factor $k = 2$ is typically selected. The measurement result, together with its uncertainty, is represented in accordance with the rules of ISO/IEC Guide 98-3:2008.

It should be noted that empirical approaches are just as valid as the modeling approach, and sometimes can lead to a more realistic evaluation of uncertainty. In fact, empirical approaches are substantially based on experimental data and long-term experience, thus reflecting the conventional practice.

Results

The statistical model, forming the basis of uncertainty evaluation methods, can be written in the form of Eq. (1) of ISO 21748:2017:

$$R_m = R + \xi_{R_m} + \sum c_i x_i' + g_{R_m} + e_{R_m}, \quad (1)$$

where

- R_m is the measurement result, for which it is assumed that it can be calculated by the corresponding function ($R_m = \frac{P_{\max}}{F_0}$, $P_{\max}$ is the maximum force preceding the rupture of the sample, F_0 is the initial cross-sectional area);
- R is the (unknown) expected value of ideal results;
- ξ_{R_m} is the bias due to traceability;
- x_i' is excursion x_i;
- c_i is the sensitivity factor equal to $\frac{\partial R_m}{\partial x_i}$;
- g_{R_m} is rounding of the measurement result according to GOST 1497-84;
- e_{R_m} is a random component of measurement uncertainty under repeatability conditions.

It was assumed that x_i' are normally distributed with a zero expected value and variance $u^2(x_i)$.

Given the model described by Eq. (1), the uncertainty of measuring R_m was evaluated using Eq. (2):

$$\begin{aligned} u^2(R_m) &= u^2(\xi_{R_m}) + \mathrm{c}^2(\mathrm{P}_{\max})u^2(\mathrm{P}_{\max}) + \mathrm{c}^2(d_0)u^2(d_0) + u^2(g_{R_m}) + u^2(e_{R_m}) \\ &= u^2(\xi_{R_m}) + \left(\frac{4}{\pi d_0^2}\right)^2 u^2(\mathrm{P}_{\max}) + \left(-\frac{8\mathrm{P}_{\max}}{\pi d_0^3}\right)^2 u^2(d_0) + u^2(g_{R_m}) + u^2(e_{R_m}), \end{aligned} \quad (2)$$

where

- $u^2(\xi_{R_m})$ is the uncertainty caused by the uncertainty of the estimate obtained based on the measurements of the reference material with the certified value;
- $u^2(x_i')$ is the uncertainty corresponding to x_i';
- $u^2(g_{R_m})$ is the uncertainty due to rounding of the measurement result;
- $u^2(e_{R_m})$ is the random component of the uncertainty of measuring the reference material under repeatability conditions.

The uncertainty, corresponding to the bias due to traceability, can be given by Eq. (3):

$$u^2(\xi_{R_m}) = u^2(R_{mGSO}) + \frac{(R_{mGSO} - \underline{R}_m)^2}{3}, \quad (3)$$

where $u^2(R_{mGSO})$ is the uncertainty corresponding to the certified value R_{mGSO} used to assess the correctness in a joint study.

If a reference material was tested n times (at least 3 times), the recommended procedure for evaluating the limits of $u^2\left(e_{R_m}\right)$ is as follows:

(a) determination of the mean $\underline{R}_m$ and the standard deviation $s_{\underline{R}_m}$;
(b) determination of the confidence interval of the mean according to formula (4):

$$u\left(e_{R_m}\right) = \frac{s_{\underline{R}_m} t(P, f)}{\sqrt{n}}, \tag{4}$$

where

t is Student's coefficient; P is the confidence level; $f = (n - 1)$ is the number of degrees of freedom;
n is the number of measurements. For $P = 0.7$ and $n = 3$, $t = 1.386$.

Table 2 represents an example of calculating the uncertainty budget of measuring the "ultimate stress" characteristic.

Discussion

An analysis of the uncertainty budget presented in Table 2 distinguishes four equivalent contributions to the combined standard uncertainty: the RM original diameter of the parallel length of a circular test piece, rounding of the result, traceability to an approved type RM, and the random component of measurements under repeatability conditions. Let us consider each of the components individually.

The contribution from the initial RM diameter is caused by the tolerance for the diameter of the test piece, equal to 0.10 mm by GOST 1497-84. This contribution, in accordance with JCGM 106:2012 [15], describes the global risk for an object (sample) selected randomly from the production process. The decision about accounting for this contribution should be taken depending on the object under assessment: an individual sample or a test procedure implemented using certain equipment in a laboratory.

The requirement of GOST 1497-84 to account for the contribution from the rounding procedure, equal to 10 N/mm^2, is apparently related to the past practice of applying tensile testing machines based on analog signals, which exhibit insufficient sensitivity to dynamic force variations during testing. When contemporary tensile testing machines based on digital signals are used, this contribution can be neglected.

The contribution from the random component of RM measurements under the repeatability conditions typically describes the quality of the operator's work and random effects, occurring during the test procedure in a laboratory using certain equipment, since the material heterogeneity is already taken into account in RM

Table 2 Uncertainty budget of measuring the "ultimate strength" characteristic

Source of uncertainty	Designation	Formula	Applied variables, parameters	Example
Original diameter of the parallel length of a circular test piece	$u(d_0)$ $c(d_0)$	$u(d_0) = \frac{\Delta_{d_0}}{\sqrt{3}}$ $c(d_0) = -2\frac{R_m}{d_0}$ $c(d_0)u(d_0)$	Table 8 GOST 1497-87 $\Delta_{d_0} = \pm 0.10$ mm $R_m = 570$ N/mm^2 $d_0 = 10.06$ mm	$u(d_0) = \frac{0.10}{\sqrt{3}} = 0.0577$ $c(d_0) = -2\frac{570}{10.06} = -113$ $c(d_0)u(d_0) = 113 \cdot 0.0577 = 6.52$ N/mm^2
Maximum force	$u(P_{max})$ $c(P_{max})$	$u(P_{max}) = \frac{\Delta_{R_m}}{\sqrt{3}}$ $c(P_{max}) = \frac{R_m}{P_{max}}$ $c(P_{max})u(P_{max})$	Limit of the permissible relative error of the force measurement $\frac{\Delta P}{P} = \pm 0.5\%$ $R_m = 570$ N/mm^2 $P_{max} = 45120$ N	$P_{max} = 45120$ N $u(P_{max}) = \frac{45120 \cdot 0.005}{\sqrt{3}} = 130$ $c(P_{max}) = \frac{570}{45120} = 0.0126$ $c(P_{max})u(P_{max}) = 130 \cdot 0.0126 = 1.64\frac{N}{mm^2}$
Result rounding	$u(g_{R_m})$	$u(g_{R_m}) = \frac{g_{R_m}}{\sqrt{12}}$	Table 2 GOST 1497-87 $g_{R_m} = 10$ N/mm^2 for $\sigma > 500$ N/mm^2	$u(g_{R_m}) = \frac{10}{\sqrt{12}} = 2.88$ N/mm^2

(continued)

Table 2 (continued)

Source of uncertainty	Designation	Formula	Applied variables, parameters	Example
Mean and MSE of CRM measurements	$\underline{R}_m, s_{\underline{R}_m}$	$\underline{R}_m = \frac{\sum_{i=1}^{n} \underline{R}_{mi}}{n} = \frac{1}{3}\sum_{i=1}^{3} \underline{R}_{mi}$ $s_{\underline{R}_m} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(\underline{R}_{mi} - \underline{R}_m)^2}$ $= \sqrt{\frac{1}{2}\sum_{i=1}^{3}(\underline{R}_{mi} - \underline{R}_m)^2}$	$\underline{R}_{mi}$—single measurement of a GSO n—number of GSO measurements ($n = 3$)	Measurement results (**465; 470; 475**) N/mm^2 $\underline{R}_m = \mathbf{470}$ N/mm^2 $s_{\underline{R}_m} = \sqrt{\frac{1}{2}(5^2 + 0 + 5^2)} = 5$ N/mm^2
Random component of the uncertainty of measuring a GSO under repeatability conditions	$u(e_{R_m})$	$u(e_{R_m}) = \frac{s_{\underline{R}_m} t(P,f)}{\sqrt{n}} = \frac{s_{\underline{R}_m} t_{0.7,2}}{\sqrt{3}}$	$t_{0.7,2} = 1.386$ for $n = 3$	$u(e_{R_m}) = \frac{5 \cdot 1.386}{\sqrt{3}} = 4$ N/mm^2

(continued)

Table 2 (continued)

Source of uncertainty	Designation	Formula	Applied variables, parameters	Example
Certified GSO value and its uncertainty (Based on the GSO certificate)	R_{mGSO} $u(R_{mGSO})$	$u(R_{mGSO}) = \frac{U(R_{mGSO})}{2}$	$R_{mGSO} =$ **446** N/mm^2 $U(R_{mGSO}) =$ 6 N/mm^2 is the value of the expanded uncertainty of the GSO according to the certificate	$u(R_{mGSO}) = \frac{6}{2} = 3$ N/mm^2
Traceability to GSO	$u(\xi_{R_m})$	$u(\xi_{\sigma_{inst}}) = \sqrt{u^2(R_{mGSO}) + \frac{(R_{mGSO} - R_m)^2}{3}}$		$u(\xi_{R_m}) = \sqrt{3^2 + \frac{\mathbf{(446-470)^2}}{3}} =$ **14.2** N/mm^2
Combined standard uncertainty	$u(R_m)$	$u(R_m) =$ $\sqrt{c^2(P_{max})u^2(P_{max}) + c^2(d_0)u^2(d_0) + u^2(g_{R_m}) + u^2(\xi_{R_m}) + u^2(e_{R_m})}$		$u(R_m) =$ $\sqrt{6.52^2 + 1.64^2 + 2.88^2 + 4^2 + \mathbf{14.2}^2} =$ 16.0 N/mm^2
Effective number of the degrees of freedom	ν_{eff}	$\nu_{eff} = \frac{u^4(R_m)}{\frac{u^4(P_{max})}{\nu(P_{max})} + \frac{u^4(d_0)}{\nu(d_0)} + \frac{u^4(g_{R_m})}{\nu(g_{R_m})} + \frac{u^4(\xi_{R_m})}{\nu(\xi_{R_m})} + \frac{u^4(e_{R_m})}{\nu(e_{R_m})}}$	$\nu(P_{max}) = \infty$ $\nu(d_0) = \infty$ $\nu(g_{R_m}) = \infty$ $\nu(\xi_{R_m}) = 2 - 1 = 1$ $\nu(e_{R_m}) = 3 - 1 = 2$	$\nu_{eff} = \frac{\mathbf{16.0^4}}{0+0+0+\frac{\mathbf{14.2^4}}{1}+\frac{4^4}{2}} = \frac{\mathbf{65536}}{\mathbf{40787}} = \mathbf{1.6}$
Expanded uncertainty	$U(R_m)$	$U(R_m) = t_{0.95,\nu_{eff}} \cdot u(R_m)$	$t_{0.95,\nu_{eff}} = 4.303$	$U(R_m) = 4.303 \cdot 16.0 = 69$ N/mm^2

metrological characteristics. In the considered example, the estimate of the contribution is equal to 4 N/mm^2. It is assumed that it is necessary to evaluate the random component for each "operator-tensile machine" system.

The contribution from traceability to the RM includes a systematic component, connected with the implementation of the test procedure in a laboratory, including due to the algorithms of tensile testing machine software, and RM heterogeneity. The main affecting factors can be preparatory operations prior to testing (resetting to zero of force and strain sensors, sample fastening type, preliminary loading value), assigned test conditions (deformation rate or loading rate), calculation errors, including in the algorithms of built-in software [8]. The testing rate used in practice is typically close to the maximum values, permitted by the test procedure, since it is necessary to conduct the maximum number of tests per one operator's work shift. However, the following implicit assumptions are ignored. On the one hand, GOST 1497-84 is a static tensile test method, i.e., reliable results are obtained at a minimum testing rate. On the other, the calibration of tensile testing machines is performed in the static mode, while tests are conducted in the dynamic mode. Therefore, the recorded values of force and elongation may have a systematic error associated with the testing rate. In the analyzed example, the contribution due to traceability is equal to **14.2** N/mm^2. It is assumed that the systematic error should be evaluated for each existing tensile testing machine.

The expanded uncertainty in the example has a value of 69 N/mm^2, which is due to the effective number of the degrees of freedom $\nu_{eff} = 1.6$. A reduction in the effective number of the degrees of freedom can be achieved using two approaches. The first is to reduce the systematic component by varying the testing rate. The second can be implemented given that the random component is not less than the contribution from traceability to a GSO.

Conclusion

The use of an approved RM as a comparison base represents one of the basic tools for ensuring traceability and controlling the accuracy of mechanical property measurements. The approach based on uncertainty budgeting for the measurement Eq. (1) can be used by laboratories to properly assess the uncertainty of the results of measuring mechanical property characteristics, taking into account the combined effect of all uncertainty sources, as well as the bias due to traceability.

The practical significance of the study lies in the possibility of applying the modeling approach of theoretical concepts based on ISO/IEC Guide 98-3:2008 and calculation algorithms provided in ISO 21748:2017 (Eq. 1) by accredited laboratories when evaluating uncertainty according to clause 7.6 of GOST ISO/IEC 17025-2019 [16].

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