METROLOGY, QUALITY, STANDARTIZATION AND CERTIFICATION

EVALUATION OF MICROHARDNESS UNCERTAINTY COMPONENTS OF COMPOSITE MATERIAL

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Abstract. The study substantiates the necessity of evaluating measurement uncertainty in the context of product quality control. The evaluation of uncertainty in determining measurement accuracy provides an evidence-based approach to verifying whether the measured quality characteristic complies with established standards. The process of uncertainty evaluation in quality control is complex and labor-intensive, involving the identification of uncertainty sources, detection of correlations among input quantities, determination of probability distribution laws of influencing factors, calculation of sensitivity coefficients, as well as standard, total and expanded uncertainties. The paper presents a methodology for determining the uncertainty components in the measurement of microhardness of structural components in a composite material. Using the Ishikawa diagram, the primary sources of uncertainty were identified, including inaccuracies in the applied load, errors in the optical system, deviations in indentation diagonal measurements, and time measurement errors. The study includes an analysis of the influencing factors and provides mathematical models for estimating standard and expanded uncertainties. The proposed methodology holds practical value for testing and research laboratories engaged in the examination of mechanical properties of materials. Measurement uncertainty evaluation enables compliance with established standards, as well as control and improvement of measurement accuracy and reliability.

Key words: uncertainty, measurement, microhardness, uncertainty evaluation, sources of error, accuracy, composite material.

1. Introduction

The uncertainty calculation is crucial across various industries where measurements and data analysis are performed. Uncertainty indicates the range of values within which the true value of a measured quantity is likely to lie, allowing for an assessment of the reliability and credibility of the results obtained [1–4]. The uncertainty evaluation enables the identification of error sources, and the optimization of measurement processes and supports the evaluation of risks associated with the application of measurement results.

The presentation of measurement, control, and testing results in the form of measurement uncertainty has become a standard on the international stage [5–7]. International standards such as ISO/IEC 17025 [8] require laboratories to evaluate measurement uncertainty during calibrations, testing, and certification procedures. This is necessary for accreditation and ensuring international recognition of results.

It should also be noted that uncertainty evaluation is a key tool for ensuring the quality of products and services. It makes it possible to determine whether the measurement results comply with established requirements.

Therefore, the uncertainty evaluation is an integral part of high-quality measurement and data analysis processes, ensuring the reliability, accuracy, and comparability of results.

2. Objective

The objective of this article is to develop a methodology for evaluating measurement uncertainty and its components for the calculation of the microhardness of structural constituents in a composite material.

3. Methodology for the uncertainty calculating of microhardness structural components of a composite material

Objective: determination of the microhardness of the composite material

Essence of the method

A diamond indenter in the shape of a regular pyramid with a square base and a specified angle between opposite faces at the apex is pressed into the surface of the sample. After removing the applied force, the lengths of the diagonals of the resulting impression are measured (Fig. 1).

Microhardness is proportional to the ratio of the numerical value of the applied force to the numerical value of the sloped surface area of the impression, which forms a regular pyramid with a square base and an apex angle equal to that of the indenter. The measurement procedure is presented as a block diagram (Fig. 2).

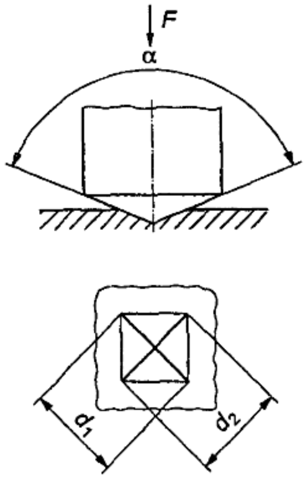

Fig. 1. Method for determining microhardness: α – angle between opposite faces at the apex of the diamond pyramidal indenter; F – applied force; d_1, d_2 – arithmetic mean of the two diagonal lengths [9]

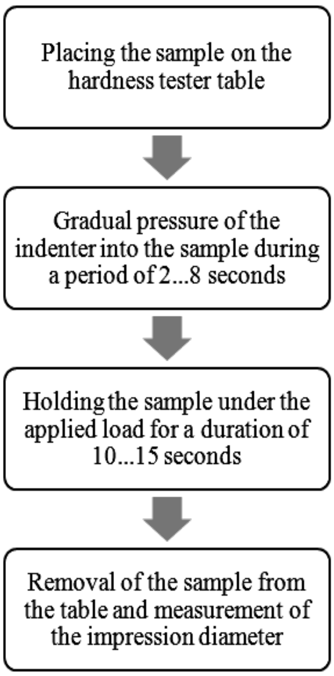

Fig. 2. Block diagram of microhardness measurement

Determination of the measured quantity

Microhardness (MPa) is calculated using the following formula:

$$H_\mu = \frac{P}{F} \cdot 9.8 = \frac{2P\sin\frac{\alpha}{2}}{d^2} \cdot 9.8 \text{ [MPa]}, \qquad (1)$$

where P – applied force, [g]; α – angle at the apex of the diamond pyramid; F – lateral surface area of the impression, [µm²]:

$$F = d^2, \text{ [µm}^2\text{]}, \qquad (2)$$

where d – length of the diagonal, [µm]:

$$d = C \cdot N, \qquad (3)$$

C – scale division value, [µm]; N – length of the diagonal in scale divisions.

Detection of uncertainty sources

The aim of this stage is to identify the main sources of uncertainty and assess their impact on the measured quantity and its inaccuracy. This is one of the most challenging stages in the process of uncertainty evaluation in measurements, as there is a risk of both neglecting certain sources of uncertainty and double-counting them [10].

To identify the main and additional sources of uncertainty, let us consider the requirements for measurement execution specified in EN ISO 6507-1 [9].

The tests should be carried out on a flat and smooth surface. The supporting surface must be clean and free from foreign substances (scale, grease, dirt, etc.). The cleanliness of the surface processing should ensure accurate measurement of the diagonal lengths of the impression. It is important that the sample rests securely on the supporting surface to avoid shifting during testing.

The indenter is brought to the sample until it touches the surface, and force is applied in a direction perpendicular to the sample's surface, without hit or vibration, until the applied force reaches the set value. The duration of the force application from the start until the full test force is reached should be no less than 2 seconds and no more than 8 seconds. When determining hardness at low force values and determining microhardness, the maximum duration should not exceed 10 seconds.

The indenter must make contact with the sample at a speed ranging from 15 µm/s to 70 µm/s during microhardness determining. The force application duration should be from 10 seconds to 15 seconds.

The thickness of the test sample or layer should be at least 1.5 times the length of the diagonal of the impression [9].

Typically, tests are conducted at room temperature, within the range of 10 °C to 35 °C. The controlled conditions tests should be carried out at a temperature of (23 ± 5) °C. During testing, the instrument must be protected from hits or vibrations.

The distance between the center of the impression and the edge of the sample should be at least 2.5 times the length of the diagonal of the impression. The distance between the centers of adjacent impressions should be at least three times the average length of the diagonals of the impressions. If two adjacent impressions have different sizes, the distance between them should be determined based on the average length of the diagonals of the larger impression.

The arithmetic mean of two measurements should be taken during calculating microhardness. For flat surfaces, the difference between the lengths of the two diagonal impressions should not exceed 5 %. If the difference is greater, it should be noted in the test report.

The uncertainty sources inherent in the microhardness measurement process are reflected in the Ishikawa diagram (Fig. 3).

The error in applied load force of the hardness tester and the optical system of the microscope are among the primary sources of uncertainty in microhardness measurement. Analysis of the testing methodology revealed that the primary sources of uncertainty are influenced by secondary factors, which signifycantly contribute to the total uncertainty of the method.

Based on the presented data, an analysis was conducted to evaluate the level of the main sources of uncertainty.

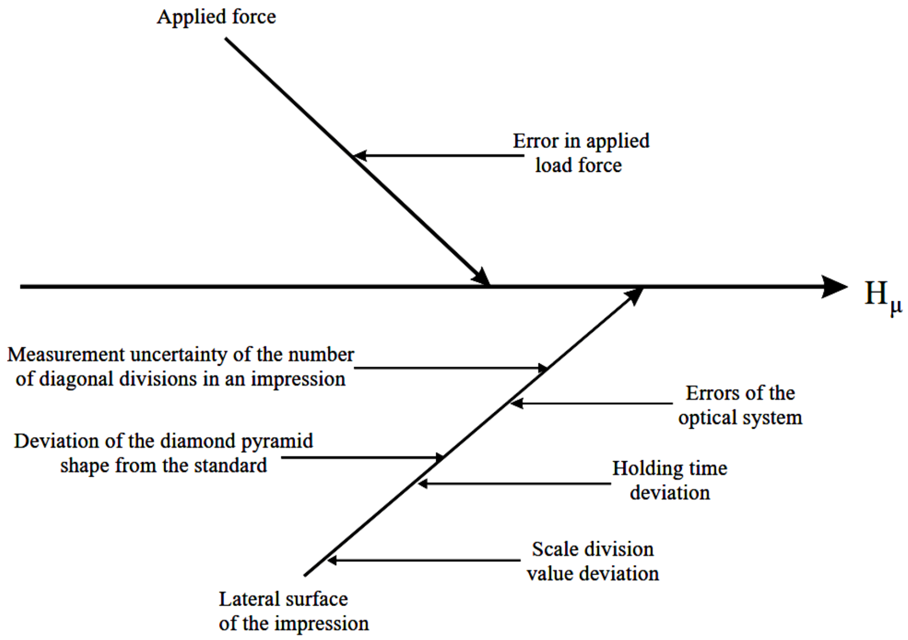

Fig. 3. Ishikawa "Cause-and-Effect" Diagram

At first it is necessary to consider the measurement model when identifying the uncertainty sources. All parameters included in the model can be potential sources of uncertainty. Moreover, quantities that are not explicitly included in the measurement model but affect the measurement result (e. g., deviations of the diamond pyramid shape from the standard, timing deviations during load application, etc.) may also act as sources of uncertainty.

The procedure for evaluating uncertainty to obtain the estimated value of the output quantity Y and the standard uncertainty $u(y)$ involves the following steps:

1) determining the best values of estimates x_i of the input quantities x_i;

2) determining the standard uncertainties of the input quantities $u(x_i)$;

3) determining the sensitivity coefficients c_i;

4) determining the total uncertainty;

5) determining the expanded uncertainty.

Contribution of the loading force uncertainty (Δ_P) *to the total uncertainty*

Special weights shaped as slotted disks with masses of 5, 10, 20, 50, 100, and 200 g are used to apply the load. The weights are placed on a designated platform located between flat springs supporting the indenter shaft. Permissible deviations in applied load are as follows: for loads up to 10 g – not more than ±0.1 g; for loads above 10 g – not more than ±1 %. As a first approximation, a rectangular probability distribution is assumed [4], and the standard uncertainty is determined according to type B evaluation [3]. In the case of a rectangular (uniform) distribution, the standard uncertainty is calculated using the formula [7]:

$$u_B = \frac{a}{\sqrt{3}}, \quad (4)$$

where a is the half-width of the confidence interval.

Contribution of deviations of the diamond pyramid shape to the total uncertainty (Δ_{pyr})

The working faces of the diamond indenter must be carefully polished and free of cracks or scratches. The shape of the working part of the diamond must conform to a perfect four-sided pyramid with an angle of 136° between opposing faces at the apex, with a maximum allowable deviation of ±20′. The edges and the apex of the pyramid must be free of rounding or crumbling.

In this case, the standard uncertainty is determined based on the assumption of a triangular probability density distribution [1]:

$$u_B = \frac{a}{\sqrt{6}}, \quad (5)$$

where a is the half-width of the confidence interval.

A triangular distribution is chosen because nominal deviations from the standard are more probable than extreme values. Therefore, the probability distribution is better approximated by a triangular distribution rather than a rectangular one.

Contribution of the holding time measurement to the total uncertainty (Δ_τ)

According to the Vickers hardness testing methodology [9], the duration of load application and the holding time during testing are clearly specified and significantly influence the obtained microhardness values. It is essential to strictly adhere to the specified test duration. The time is controlled with an accuracy of ±0.5 seconds. Assuming that the distribution within the specified range has a triangular shape, the standard uncertainty is calculated by dividing the time variation by $\sqrt{6}$ according to formula (5) [7].

Contribution of optical system errors to the total uncertainty (Δ_{opt}).

Errors related to the imperfections of the microscope's optical system at a fixed magnification of the objective and eyepiece include:

1) backlash of the eyepiece micrometer screw;

2) unevenness of the eyepiece micrometer screw pitch;

3) misalignment between the screw axis and the diagonal direction of the impression;

4) aperture diaphragm;

5) focusing of the objective and eyepiece (parallax);

6) condenser and field diaphragm (direction and intensity of illumination).

The magnitude of these errors and the variability of the values associated with the imperfections of the optical system primarily depend on the operator's experience. It is theoretically impossible to calculate these errors precisely, therefore, experimental data established that the relative error of diagonal measurement of the impression depends on the diagonal length and constitutes at a length of 20 μ – 5 %, 10 μ – 10 % and 5 μ – 20 %. The standard uncertainty is calculated using formula (4), assuming a rectangular distribution within the specified limits [7].

Contribution of determining the number of scale divisions on the impression diagonal to the total uncertainty (Δ_N)

In the presence of a set of experimental data, the uncertainty due to repeated measurements of the number of scale divisions of the impression diagonal is evaluated as Type A [7], using formula [6]:

$$u_A = \sqrt{\frac{1}{n(n-1)} \cdot \sum_{i=1}^{n} (X_i - \bar{X})^2}, \quad (6)$$

where X_i is the i-th measurement result; $\bar{X}$ – is the arithmetic mean of the measurement results; n is the number of observations.

Contribution of the scale division error to the total uncertainty (Δ_C)

The uncertainty associated with the scale division error of the eyepiece micrometer is evaluated using the calibration certificate data and manufacturer documentation. According to these, measurements with the eyepiece micrometer can be performed with an accuracy of ±0.5 scale divisions. The scale division value in our case was 0.25 µm. The standard uncertainty, in this case, is calculated based on the assumption of a triangular probability density distribution using formula (5), as nominal deviation values are more probable than extreme ones.

Measurements were carried out under normal conditions and in the absence of vibrations; therefore, standard uncertainties caused by these factors were not considered.

Thus, the measurement function (model) in our case takes the form:

$$H_\mu = f\left(\Delta_P,\ \Delta_{opt},\ \Delta_{pyr},\ \Delta_N,\ \Delta_C,\ \Delta_\tau\right), \quad (7)$$

where Δ_P – load force error, [g]; Δ_{opt} – optical system error, [µm]; Δ_{pyr} – deviation of the diamond pyramid shape from the standard, [°]; Δ_N – error in determining the number of divisions of the impression diagonal; Δ_C – scale division error, [µm]; Δ_τ – timing measurement error, [s].

Quantitative expression of uncertainty components

At this stage, the uncertainty from each source identified during the uncertainty source identification phase must be expressed quantitatively and then converted into standard uncertainty.

$$u(\Delta_P) = \frac{\Delta_P}{\sqrt{3}} = \frac{1}{\sqrt{3}} = 0.577 \text{ g}$$

$$u(\Delta_{opt}) = \frac{\Delta_{opt}}{\sqrt{3}} = \frac{0.541}{\sqrt{3}} = 0.312 \text{ µm}$$

$$u(\Delta_{pyr}) = \frac{\Delta_{pyr}}{\sqrt{6}} = \frac{20'}{\sqrt{6}} = 0.135°$$

$$u(\Delta_C) = \frac{\Delta_C}{\sqrt{6}} = \frac{0.125}{\sqrt{6}} = 0.051 \text{ µm}$$

$$u(\Delta_\tau) = \frac{\Delta_\tau}{\sqrt{6}} = \frac{0.5}{\sqrt{6}} = 0.204 \text{ s}$$

$$u_A(N) = \sqrt{\frac{1}{n(n-1)} \cdot \sum_{i=1}^{n} (N_i - \bar{N})^2} = 0.3$$

All the above-mentioned factors contribute to the combined uncertainty of measuring the number of scale divisions along the diagonal of the impression, which is determined using the following formula:

$$\frac{u_c(N)}{N} = \sqrt{\left(\frac{u_A(N)}{N}\right)^2 + \left(\frac{u(\Delta_C)}{N}\right)^2 +} $$
$$+\sqrt{\left(\frac{u(\Delta_{opt})}{N}\right)^2 + \left(\frac{u(\Delta_{pyr})}{\alpha}\right)^2 + \left(\frac{u(\Delta_\tau)}{\tau}\right)^2} \quad (8)$$

Calculations showed that $\frac{u_c(N)}{N}$ is equal to 0.024.

Then, $u_c(N) = N \cdot 0.024 = 1.018$.

The intermediate results obtained during the implementation of the basic algorithm are conveniently presented in the form of an uncertainty budget (Table 1), which includes a list of all input quantities, their estimates along with the associated standard measurement uncertainties, and the type of distribution.

In addition to the information about the input quantities, it is convenient to include information about the measured quantity in the uncertainty budget: measurement result, total standard uncertainty, effective degrees of freedom, coverage factor, and expanded uncertainty.

Calculation of total standard uncertainty

The microhardness of a composite material is determined using the formula:

$$H_\mu = \frac{2P\sin\frac{\alpha}{2}}{(C\cdot N)^2} \cdot 9.8 \text{ [MPa]} \quad (9)$$

The values of the input quantities, their standard uncertainties, and relative standard uncertainties are presented in Table 2.

Table 1. Uncertainty budget of microhardness components during testing

Name of input quantity	Symbol	Type of uncertainty evaluation	Type of distribution	$\frac{u(\Delta x_i)}{\Delta x_i}$	Contribution to total uncertainty, %
Error of load force	Δ_P	B	rectangular	0.0058	9.8
Error of the optical system	Δ_{opt}	B	rectangular	0.0072	12.2
Deviation of the diamond pyramid shape from the norm	Δ_{pyr}	B	triangular	0.001	1.7
Error in determining the number of scale divisions along the diagonal of the impression	Δ_N	A	normal	0.0235	39.7
Error in the scale division value	Δ_C	B	triangular	0.0012	2
Error of time measurement	Δ_τ	B	triangular	0.0204	34.6

Table 2. Input quantities and their uncertainties

Quantity	Value *x*	$u(x_i)$	$u(x_i)/x_i$
Load force	100 g	0.577 g	0.006
Angle at the apex of the diamond pyramid	136°	0.135°	0.001
Scale division value	0.25 μm	0.051 μm	0.001
Number of scale divisions along the diagonal of the impression	43.3	1.018	0.024

Using these values, the microhardness of the composite material is determined as:

$$H_\mu = 15505.29 \text{ MPa}.$$

The correlation between the input quantities is identified to determine the total uncertainty. In our case, there is a correlation between the applied load force (P) and the number of scale divisions along the diagonal of the impression (N). Considering that two of the input quantities are correlated, the total standard uncertainty is determined using the expression [7]:

$$u_c^2(H_\mu) = \sum_{i=1}^{n} c_i^2 u^2(x_i) +,$$
$$+2\sum_{i=1}^{n-1}\sum_{j=i+1}^{n} c_P c_N u(P)u(N)r(P,N), \quad (10)$$

where $u(x_i)$ – is the standard uncertainty of the *i*-th input quantity; $r(P,N)$ – the degree of correlation between P and N; c_P and c_N – sensitivity coefficients; *n* – number of input quantities.

The sensitivity coefficients, defined as $c_i = \frac{\partial\varphi}{\partial x_i}$, indicate how the output estimate φ changes with variations in the input estimates $x_1, \ldots x_n$ are given by [4]:

$$c_P = \frac{\partial\left(\frac{2P\sin\frac{\alpha}{2}}{(C\cdot N)^2}\cdot 9.8\right)}{\partial P} = \frac{2\cdot 9.8\cdot\sin\frac{\alpha}{2}}{C^2\cdot N^2}, \quad (11)$$

$$c_N = \frac{\partial\left(\frac{2P\sin\frac{\alpha}{2}}{(C\cdot N)^2}\cdot 9.8\right)}{\partial N} = \frac{-2\cdot 9.8\cdot 2\cdot P\cdot\sin\frac{\alpha}{2}}{C^2\cdot N^3}, \quad (12)$$

In our case, the sensitivity coefficients were as follows: $c_P = 155.053$ and $c_N = -716.18$.

The degree of correlation between x_P and x_N is characterized by the correlation coefficient, which is determined based on the following formula:

$$r(P,N) = \frac{u(P,N)}{u(P)u(N)}. \quad (13)$$

The covariance estimate of two correlated input quantities P and N with estimates $\bar{P}$ and $\bar{N}$, obtained from repeated observations, is calculated using the following formula [1]:

$$u(P,N) = s(\bar{P},\bar{N}), \quad (14)$$

where $s(\bar{P},\bar{N})$ is determined using the expression:

$$s(\bar{P},\bar{N}) = \frac{1}{n(n-1)}\sum_{i=1}^{n}(P_i - \bar{P})(N_i - \bar{N}), \quad (15)$$

where P_i and N_i – are the measurement results of quantities P and N respectively, and $\bar{P}, \bar{N}$ – are their mean values.

In our case, when calculating $s(\bar{P}, \bar{N})$ using the difference $(P_i - \bar{P})$ we accept the absolute error of the loading force. In our case, the absolute error of the loading force is 1 g.

Therefore,

$$u(P,N) = s(\bar{P}, \bar{N}) = 3.158 \cdot 10^{-16}.$$

$$r(P,N) = \frac{u(P,N)}{u(P)u(N)} = \frac{3.158 \cdot 10^{-16}}{0.577 \cdot 1.018} = 5.37 \cdot 10^{-16}.$$

Therefore, the total standard uncertainty in determining the microhardness of the composite material is:

$$u_c^2(H_\mu) = c_P^2 u^2(P) +$$
$$+ c_N^2 u^2(N) + 2 c_P c_N u(P) u(N) r(P,N) =$$
$$= 155.05^2 \cdot 0.57^2 + (-716.18)^2 \cdot 1.018^2 +$$
$$+ 2 \cdot 155.05 \cdot (-716.18) \cdot 0.57 \cdot 1.018 \cdot 5.37 \cdot 10^{-16} =$$
$$= 515524,$$

$$u_c(H_\mu) = \sqrt{515524} = 718 \text{ MPa}.$$

To determine the total standard uncertainty $u_c(H_\mu)$ it is also possible to use Excel spreadsheets, as they are applicable for expressions of any complexity. The completed spreadsheet is shown in Fig. 4. The parameter values are entered in the second row from C2 to D2. Their standard uncertainties are entered in the row below (C3–D3). The values from (C2–D2) are copied to the second column of the table from B5 to B6. The result (H_μ), obtained from these values, is given in cell B8. Cell C5 gives the value of P from C2 plus its uncertainty from C3. The calculation result, which uses the values C5–C6, is shown in cell C8. Column D is filled in a similar manner. The values in row 9 (C9–D9) are formed as the difference between rows (C8–D8) minus the value specified in B8. In row 10 (C10–D10), the values from row 9 (C9–D9) are squared and summed, leading to the value specified in B10. Cell B12 gives the total standard uncertainty, which is the square root of B10.

	A	B	C	D
1			P	N
2			100	43,3
3			0,58	1,018
4				
5	P	100	100,58	100
6	N	43,3	43,3	44,318
7				
8	Hμ	15505,3	15595,2	14801,2
9	u(y,xi)		89,9307	-704,143
10	u(y)^2, u(y,xi)^2	503905	8087,53	495817
11				
12	u(Hμ)	709,862		

Fig. 4. Uncertainty calculation using spreadsheets

It should be noted that the results of calculating the total uncertainty $u_c(H_\mu)$, obtained manually and through the spreadsheet method, show almost complete agreement. The spreadsheet method is much easier and faster; however, it cannot be used independently from the previous calculations during the development of the methodology. In the future, when practically applying the methodology, spreadsheets significantly simplify the processing of measurement results and automate the calculation process.

The contributions to the total uncertainty from various factors are shown in Fig. 5.

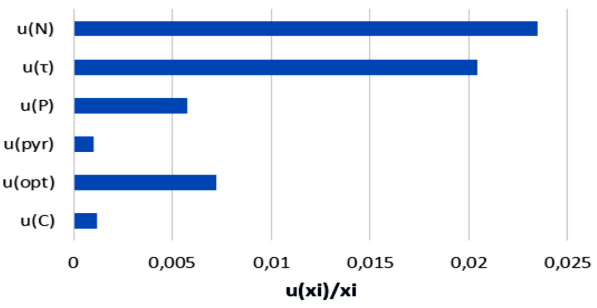

Fig. 5. Contributions of measurement uncertainty components to the total uncertainty in microhardness of composite material

An analysis of the magnitude of each component shows that the contribution related to the error in determining the number of scale divisions on the diagonal of the impression and the time measurement error during loading is undoubtedly the largest and most dominant. The significant influence of these components is associated with manual control of the measurement process, as they directly depend on the individual operator and his skills. The use of instruments with an automated measurement process will significantly improve the accuracy of microhardness measurements.

The expanded uncertainty $U(H_\mu)$ is obtained by multiplying the total standard uncertainty by the coverage factor, according to the formula [2]:

$$U(H_\mu) = k \cdot u_c(H_\mu), \tag{16}$$

where k is the coverage factor.

In the guide for expressing measurement uncertainty [7], it is recommended to use a coverage factor of 2 for a confidence level of 0.95. With k=2, the expanded uncertainty will be:

$$U(H_\mu) = 2 \cdot 718 = 1430 \text{ МПа}$$

Thus, the result of the microhardness measurement of the composite material will be expressed as:

$$H_\mu = (15505 \pm 1430) \text{ MPa}, p{=}0.95.$$

4. Conclusions

The study conducted in this work made it possible to develop an effective methodology for evaluating the components of measurement uncertainty in the microhardness of composite materials. The paper presents a measurement model that includes the primary sources of uncertainty. The calculation of each component was carried out taking into account the type of distribution and the correlation relationships. Based on the analysis of the impact of each component, it was established that the greatest contribution to the total uncertainty in the microhardness measurement comes from inaccuracies in determining the number of divisions on the diagonal of the impression and the holding time, which can be attributed to the manual control of the measurement process. The use of automated devices would signifycantly improve the accuracy of the measurements. Knowledge of measurement uncertainty enables the comparison of measurement results with established requirements during conformity evaluation, determining the probabilities of making correct decisions, and managing emerging risks while taking them into account. The obtained results contribute to enhancing the reliability of data during the investigation of the mechanical properties of materials in scientific and industrial laboratories.

Conflict of Interest

The authors declare re no financial or other potential conflicts of interest regarding this work.

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