

Methods for Accuracy Increasing of Solid Brittle Materials Fracture Toughness Determining

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Abstract

Method for determining of the fracture toughness of brittle materials by indentation is described. The critical stress intensity factor K_{IC} quantifies the fracture toughness. Methods were developed and applied to improve the accuracy of K_{IC} determination due to atomic force microscopy and nanoindentation. It is necessary to accurately determine parameters and dimensions of the indentations and cracks formed around them in order to determine the K_{IC} . Instead of classical optical and scanning electron microscopy an alternative high-resolution method of atomic force microscopy was proposed as an imaging method.

Three methods of visualization were compared. Two types of crack opening were considered: along the width without vertical displacement of the material and along the height without opening along the width. Due to lack of contact with the surface of the samples under study, the methods of optical and scanning electron microscopy do not detect cracks with a height opening of less than 100 nm (for optical) and less than 40–50 nm (for scanning electron microscopy). Cracks with opening in width are determined within their resolution. Optical and scanning electron microscopy cannot provide accurate visualization of the deformation area and emerging cracks when applying small loads (less than 1.0 N). The use of atomic force microscopy leads to an increase in accuracy of determining of the length of the indent diagonal up to 9.0 % and of determining of the crack length up to 100 % compared to optical microscopy and up to 67 % compared to scanning electron microscopy. The method of atomic force microscopy due to spatial three-dimensional visualization and high accuracy ($XY \pm 0.2$ nm, $Z \pm 0.03$ nm) expands the possibilities of using indentation with low loads.

A method was proposed for accuracy increasing of K_{IC} determination by measuring of microhardness from a nanoindenter. It was established that nanoindentation leads to an increase in the accuracy of K_{IC} determination by 16–23 % and eliminates the formation of microcracks in the indentation.

Keywords: fracture toughness, accuracy, indentation method, atomic force microscopy, nanoindentation.

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Способы повышения точности определения вязкости разрушения твёрдых хрупких материалов при индентировании

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Приведено описание метода определения вязкости разрушения хрупких материалов индентированием. Количественно вязкость разрушения характеризуется критическим коэффициентом интенсивности разрушения K_{IC} . Использование атомно-силовой микроскопии и наноиндентирования позволило разработать и применить способы повышения точности определения K_{IC} . Для определения K_{IC} необходимо точно определять параметры и размеры отпечатков индентирования и образованных вокруг них трещин. В качестве метода визуализации вместо классических оптической и сканирующей электронной микроскопий предложен альтернативный высокоразрешающий метод атомно-силовой микроскопии.

Проведено сравнение трёх методов визуализации. Рассмотрено два типа раскрытия трещин: по ширине без смещения материала по вертикали и по высоте без раскрытия по ширине. Методы оптической и сканирующей электронной микроскопий из-за отсутствия контакта с поверхностью исследуемых образцов не определяют трещины с раскрытием по высоте менее 100 нм (для оптической) и менее 40–50 нм (для сканирующей электронной микроскопии). Трещины с раскрытием по ширине определяют в рамках своей разрешающей способности. Оптическая и сканирующая электронная микроскопии не могут обеспечить точную визуализацию области деформации и формирующихся трещин при применении малых нагрузок (меньше 1,0 Н). Применение атомно-силовой микроскопии приводит к повышению точности определения длины диагонали отпечатка до 9,0 % и определения длины трещины до 100 % по сравнению с оптической микроскопией и до 67 % по сравнению со сканирующей электронной микроскопией. Метод атомно-силовой микроскопии благодаря пространственной трёхмерной визуализации и высокой точности (по $XU \pm 0,2$ нм, по $Z \pm 0,03$ нм) расширяет возможности применения индентирования с применением низких нагрузок.

Предложен способ повышения точности определения K_{IC} за счёт измерения микротвёрдости с наноиндентора. Установлено, что наноиндентирование приводит к повышению точности определения K_{IC} на 16–23 % и исключает образование микротрещин в отпечатке.

Ключевые слова: вязкость разрушения, точность, метод индентирования, атомно-силовая микроскопия, наноиндентирование.

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Introduction

Reducing of the measurement error of any physical quantity is always an urgent task. Development of new methods, use of other physical techniques allows them to be applied to existing measurement methods to improve accuracy and reduce the error in determining of physical quantities. In this work, we will present a method for measuring the physical and mechanical properties of hard brittle materials, namely, fracture toughness (or crack resistance). This parameter characterizes the limiting state of any solid material and the ability to resist crack development [1–6]. It is extremely important for various types of ceramics (carbide, oxide, nitride) [7, 8] and coatings based on them, which combine special physical-mechanical, thermophysical, bioinert, antioxidant and wear-resistant properties [2, 7]. One of the main tasks of ceramic production technology is to increase their strength, to prevent the likelihood of sudden brittle fracture, appearance of chips on the surface, or even the destruction of the part.

The critical stress intensity factor K_{IC} quantifies the fracture toughness [1, 3–6]. There are many methods to determine this characteristic (bending, torsion, rupture, etc.). However, all of them are of limited use due to the complexity or impossibility of preparing test samples with the required notch geometry and are economically unprofitable. The indentation method [4–6, 9] does not require samples of complex shape. It uses thin sections and consists in the study of the deformation area on the material surface after indentation, followed by the calculation of K_{IC} .

The purpose of the work was to improve the accuracy of determining the critical stress intensity factor K_{IC} by using the methods of atomic force microscopy and nanoindentation, to establish the influence of the visualization method of the deformation area, the calculation model, and microhardness values on the accuracy of determining K_{IC} .

Analysis of the method for determining fracture toughness by indentation

Determination of fracture toughness K_{IC} by indentation is based on the introduction of a diamond indenter in the form of a tetrahedral Vickers pyramid into the surface of the test sample (Figure 1) under a selected load depending on the material and size of the sample [3–6]. Performed at least three indentations at each load. The prints are visualized in an optical microscope (OM) or a scanning electron microscope (SEM) after indentation, the length of the print diagonals d_1 and d_2 is determined, and the print half-diagonal length $a = (d_1 + d_2)/4$ is calculated. Measured the length of the cracks (l is the length of the crack near the indent, c is the length of the crack measured from the center of the indent) near each indent, and then determine the average values of the crack lengths for the sample. Determined the physical and mechanical properties of the material (microhardness H_V and elasticity modulus E). On the ratio c/a determines the type of cracks (Palmquist cracks or median cracks) [6] around the prints after determining the values of a , l , c , H_V and E . A mathematical calculation model is selected depending on the type of cracks and the critical stress intensity factor K_{IC} is determined.

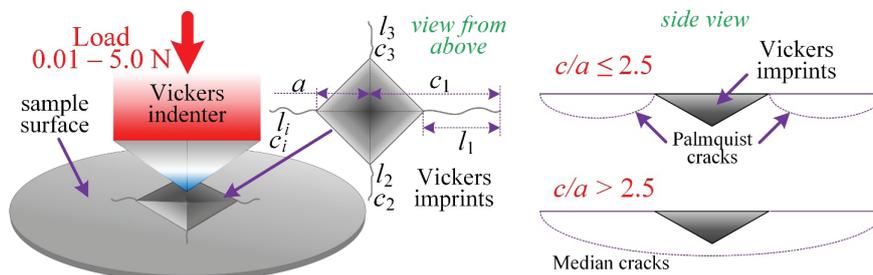


Figure 1 – Method and indentation imprint with defined parameters

Development of methods for reducing the error in determination of fracture toughness by the indentation method

Determination of fracture toughness by indentation is a computational and experimental method.

To accurately determine K_{IC} , it is important both to use mathematical calculation models (adequate to the range not only of high loads from 1.0 N and above, but also to low loads of 0.01–0.5 N) and accurate experimental characteristics of the material and the deformation region. There are factors that affect

the accuracy of determining the fracture toughness of a material by indentation. These include the following factors: the accuracy of determining the length of the diagonals of the indentation mark and the length of the cracks around it; method for determining the microhardness of a material.

Mathematical Model of Calculation. There are many mathematical models for determining the critical stress intensity factor K_{IC} [10]. In most of the models used earlier in the literature, loads of more than 1 N were used. In cases where small loads (0.25–0.75 N) were used, the values of a , l and c were determined inaccurately, and because of this, at low loads, the difference in a , l and c was not detected. Justification of the choice and determination of the correctness of mathematical models for calculating K_{IC} and their ability to maintain the stability of K_{IC} values in a wide range of loads, primarily at low loads (from 0.01 to 0.5 N), will expand the capabilities of the indentation method.

Visualization of the Deformation Area. OM and SEM are used in the classical approach to visualize indentation prints, determine the length of the diagonals of the indentation print and the length of cracks around it on the surface of the material under study. Each of these methods allows, within its resolution, to determine the linear geometric dimensions of the indentation imprint and the formed cracks.

The resolution of an optical microscope is characterized by the smallest distance between two points that are visible separately in the microscope. This distance is directly proportional to the wavelength of the light radiation incident on the object under study. The minimum dimensions of a distinguishable object are approximately equal to half the wavelength of the incident light. Standard optical microscopes use visible light. Objects around $0.25 \cdot 10^{-6}$ m or 250 nm in size can be seen under a microscope.

The resolution of the SEM is determined by the electron wavelengths and the numerical aperture of the system. The wavelength of electron radiation depends on its energy, which is affected by the accelerating voltage. The electron energy is $E = V \cdot e$, where V – potential difference, traversable by electron; e – electron charge. Thus, objects larger than 1.0–1.2 nm in size can be distinguished in the SEM.

If you change the OM and SEM measurement method to a micromechanical method – atomic force microscopy (AFM) with a vertical resolution of at least 0.03 nm, then due to spatial

three-dimensional visualization (which neither OM nor SEM has), it becomes possible to determine the depth of the indentation imprint and cracks, as well as the height of their opening [11]. In AFM the resolution depends on the radius of curvature of the tip of the used probe and the nature of its interaction with the surface of the object (contact or semi-contact) [12]. The AFM resolution in XY is limited by the capabilities of the device and is 0.2–0.5 nm.

Microhardness Definition. To determine the fracture toughness K_{IC} , it is necessary to determine the microhardness and elastic modulus of the material under study. Indentations are carried out at various loads, three to five (minimum number) of indentations at each load to determine K_{IC} . The classical method for determining fracture toughness K_{IC} uses the values of microhardness H_V determined by the Vickers method (GOST 9377–81). Microhardness H_V according to the Vickers method is determined by the formula (1) [13]:

$$H_V = 1.854 \frac{P}{d_{mean}^2}, \quad (1)$$

where d_{mean} is arithmetic mean of the diagonals of the imprint of a tetrahedral Vickers pyramid after indentation, m; P is indenter load, N.

During indentation, all the energy consists of the elastic and plastic components of the deformation and is spent on the formation of an imprint on the surface sample. The presence of cracks around the indentation indicates that part of the energy was spent on their formation in the sample, as well as the inability to reliably determine the microhardness of the sample material. Unreliable values of Vickers microhardness H_V lead to incorrect determination of K_{IC} .

An alternative method for determining microhardness can be the method of nanoindentation (NI). A Berkovich-type diamond indenter and loads of no more than 5 mN are used when measuring microhardness on a nanoindenter (ISO 14577-1:2015). The microhardness H_{Ber} by the NI method is determined by the formula [14]:

$$H_{Ber} = \frac{P_{max}}{A}, \quad (2)$$

where P_{max} is maximum indentation force of the Berkovich pyramid, N; A is resulting contact area under this load, m^2 .

Contact area A was determined by the formula [14]:

$$A = \frac{\pi}{4} \left(\frac{S}{E_r} \right)^2, \quad (3)$$

where S is unload curve stiffness; E_r is reduced module, GPa.

The use of a high-precision NI method and low loads makes it possible to exclude the formation of cracks in the material. Accordingly, the microhardness values determined by this method are correct and accurate.

Samples and equipment

To compare the visualization methods of the deformation area, diagonals of the length of the indentation print and length cracks, we used: an optical microscope MICRO-200 (JSC Planar, Republic of Belarus) and a lens with a magnification of 400×, SEM – JSM-7001F (JEOL, Japan) with resolution in secondary electrons 1.2 nm (at an accelerating voltage of 30 kV) and AFM – Dimension Fast-Scan (Bruker, USA) with XY resolution ± 0.2 nm, $Z \pm 0.03$ nm. A section of silicon carbide ceramic was used as a sample [7, 8]. Microhardness imprints on the sample were made using a PMT-3M microhardness tester (LOMO, Russia) with a Vickers tetrahedral diamond pyramid at a constant load of 1.0 and 2.0 N.

Experimental determination of the influence of the mathematical calculation model and the method for determining microhardness on the value of fracture toughness K_{IC} was carried out on several materials – single-crystal silicon wafers Si of three orientations (100), (110) and (111) (JSC “INTEGRAL”, Belarus) with a size of $\varnothing 100$ mm and a thickness of 0.5 mm, AT-cut quartz plates with a diameter of 12 mm and a thickness of 3 mm after chemical-mechanical (CMP) and magnetorheological (MRF) polishing, slide and cover glass. Indentation prints were made using a PMT-3M microhardness tester with a load from 0.01 to 5.0 N

Five indentations were performed for each load. Then visualization was carried out using AFM, the indentation parameters (d_1 , d_2) and crack length (l , c) were determined. Microhardness was determined by two methods: by Vickers using formula (1), by the NI method (load 5 mN). Microhardness H_V by the Vickers method was determined on PMT-3M. The NI method was determined using a Hysitron 750 Ubi nanoindenter (Bruker, United States) with a Berkovich-type diamond tip with a curvature radius of 60 nm [14]. Then K_{IC} was determined.

The contribution of the mathematical model of calculation to the accuracy of determining the fracture toughness

With the existing set of models for calculating K_{IC} using the indentation method, it has been established that it is impossible to correctly determine the fracture toughness of the material under study using most models, especially at low indentation loads (0.01–0.5 N). The models used for comparison are given in [15]. K_{IC} for each sample was determined at loads of 0.01–5.5 N using six formulas from [15]. It was found that the mathematical models of calculation (4) and (5) [3, 5, 6, 15] given below show the correctness and stability of the K_{IC} values in the entire range of loads (from 0.01 to 5.5 N). These models are designed and are given in [5, 6]. Unlike others, they include the parameters of the indentation imprint (diagonals length d) and the formed cracks length l and c . Also include parameters characterizing the material (microhardness H and elasticity modulus E):

$$K_{IC} = 0.048 \left(\frac{l}{a} \right)^{-\frac{1}{2}} \cdot \left(\frac{H_V}{E\Phi} \right)^{-\frac{2}{5}} \cdot \left(\frac{H_V a^2}{\Phi} \right)^{\frac{1}{5}}, \quad (4)$$

$$K_{IC} = 0.129 \left(\frac{c}{a} \right)^{-\frac{3}{2}} \cdot \left(\frac{H_V}{E\Phi} \right)^{-\frac{2}{5}} \cdot \left(\frac{H_V a^2}{\Phi} \right)^{\frac{1}{5}}, \quad (5)$$

where l is crack length near the indent, m; a is half-diagonal length, m; E is elasticity modulus, GPa; Φ is bond reaction index in the crystal lattice ($\Phi \approx 3$); H_V is Vickers hardness, GPa; c is crack length from the center of the indent, m.

In works [5, 6] in addition to models, described the conditions for their selection depending on the type of cracks (Figure 1). The condition is as follows: if $c/a \leq 2.5$, then Palmquist cracks form in the sample (Figure 1) and the calculation is carried out according to model (4), and if $c/a > 2.5$, then median cracks form in the sample (Figure 1) and the calculation is carried out by model (5) [5, 6].

According to the obtained values for each load, the average value was determined using six formulas from [15] (Table 1). The values according to formulas (4) and (5) depending on the c/a ratio were taken as the actual K_{IC} values for the test material with a standard deviation of less than 10 % [15]. Bold type in Table 1 indicates the actual K_{IC} values for each sample.

It has been established that deviations 12–74 % (Table 2) give mathematical models [15] from the actual values of fracture toughness of that depend directly on the load P (Figure 3).

Table 1

Fracture toughness and deviation from the actual value for silicon, quartz and glass

Silicon					
K_{IC} , (MPa·m ^{1/2})			Deviation of the K_{IC} value in % from the actual value		
(100)	(110)	(111)	(100)	(110)	(111)
0.59±0.11	1.10±0.55	0.74±0.42	50.9	12.4	37.8
0.74±0.22	1.06±0.54	0.86±0.15	38.0	15.7	27.5
1.20±0.05	1.26±0.09	1.19±0.10	0.0	0.0	0.0
0.38±0.07	0.55±0.32	0.46±0.33	68.1	56.4	61.5
1.23±0.07	1.24±0.08	1.20±0.08	2.5	1.6	0.8
0.31±0.13	0.45±0.21	0.39±0.22	74.3	64.3	67.2
Quartz					
CMP		MRF		MRF	
1.89±0.89		1.15±0.51		29.8	17.5
1.78±0.77		1.57±0.69		22.0	11.9
1.46±0.12		1.40±0.17		0.0	0.0
0.95±0.38		0.88±0.38		34.7	37.3
1.54±0.13		1.33±0.15		5.3	4.7
0.58±0.23		0.46±0.21		60.0	67.2
Glass					
Slide		Cover		Cover	
1.97±0.47		1.67±0.08		37.48	51.49
2.15±0.04		1.88±0.09		50.25	70.76
1.42±0.03		1.10±0.05		0.0	0.0
1.06±0.30		0.92±0.04		25.88	16.03
1.39±0.01		1.11±0.05		2.46	0.91
0.60±0.17		0.49±0.02		57.70	55.67

Comparison of visualization methods and the accuracy of determining the diagonals of the imprint of length and cracks

Visualization of the deformation area (or indentation imprint) was carried out on OM, SEM and AFM after applying marks (Figure 2). Comparison of the quality and accuracy of imaging compared on a specific selected imprint. On optical images (Figure 2a, b), the selected indentations are marked with a red square.

These imprints were then visualized using SEM and AFM (Figure 2). It is very difficult to accurately determine the presence of cracks around the imprint and their length from optical images. The length d_1 and d_2 of the imprints is determined approximately (Table 2). SEM images do not always clearly show the borders of the imprint (Figure 2a). Diagonal length cannot be determined exactly. When comparing the determination of the length of the indent diagonals (d_1 and d_2), it was found that AFM

allows increasing the accuracy of determining the length of the diagonal up to 9 % compared to OM

and up to 2.3 % compared to SEM, if scanning fields of $10 \times 10 \mu\text{m}^2$ – $50 \times 50 \mu\text{m}^2$ are used.

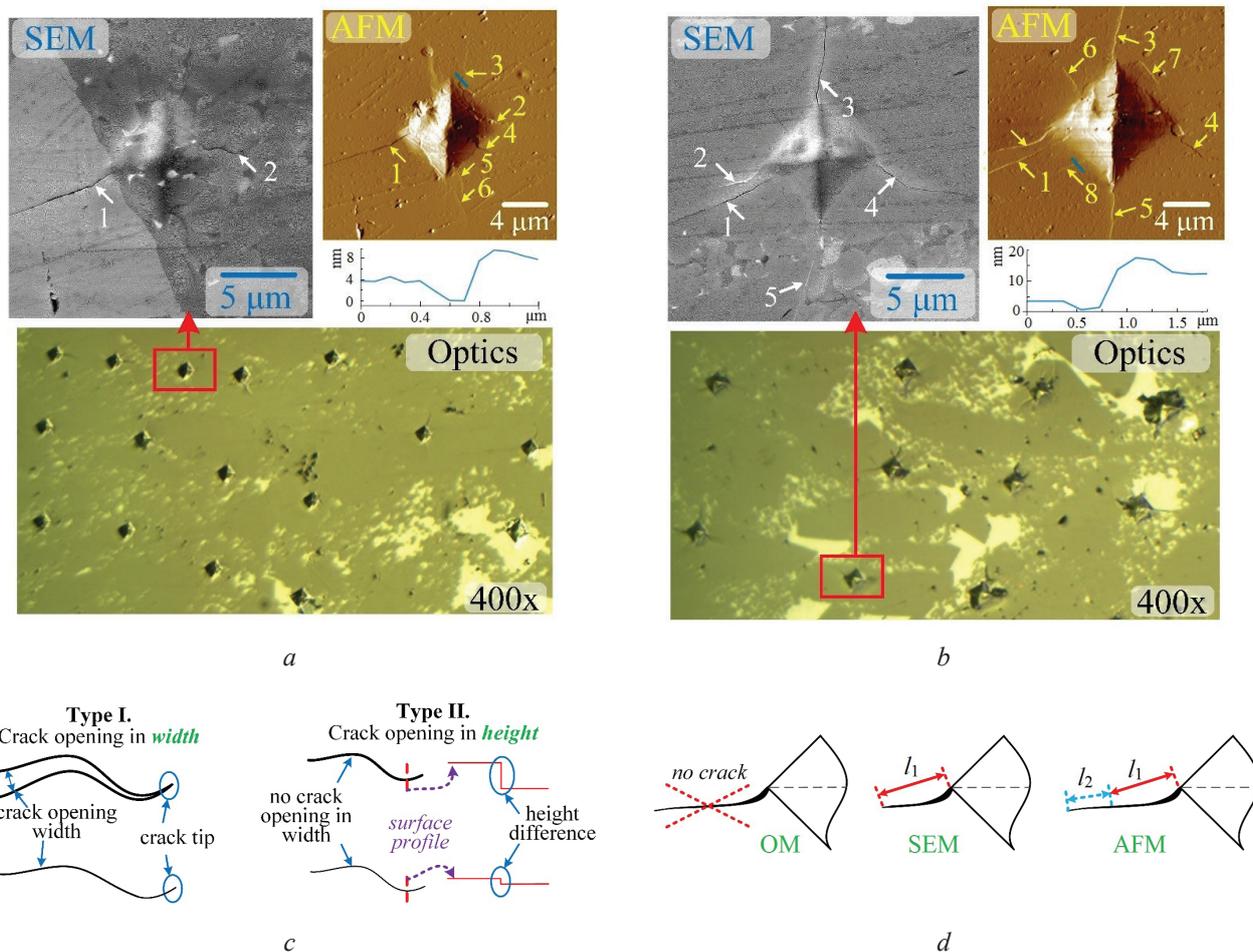


Figure 2 – Optical (400×), images from scanning electron and atomic force microscopy of imprints (*a*, *b*), features of crack opening (*c*) and influence on the accuracy of determining their length (*d*): *a* – 1.0 N; *b* – 2.0 N; *c* – two types of crack opening; *d* – determination of *l* crack (using the example of crack No. 2 in Figure 2*b*)

Table 2

Indentation diagonal length and percentage difference compared to optical microscopy

	OM		SEM		%		AFM		%	
	1.0 N	2.0 N								
P, N	1.0 N	2.0 N								
$d_1, \mu\text{m}$	9.97	10.61	9.00	11.25	9.7	6.0	9.19	11.52	7.8	8.6
$d_2, \mu\text{m}$	10.07	11.54	9.20	11.35	8.6	1.6	9.24	11.37	8.2	1.5

During indentation, two types of crack opening are formed: the first type is widthwise opening, the second type is vertical opening (Figure 2*c*). Cracks of the first type open in the *XY* plane without vertical displacement of the material. Cracks of the

second type open along the *Z* axis with little or no width opening (Figure 2*c*). OM and SEM detect cracks of the first type (in width) only within their resolution. OM reveals cracks with a width opening of at least 250 nm, SEM – at least 1–1.2 nm. Cracks

of the second type (with opening) are either not detected by OM and SEM, or they are determined, but not the entire length: the height difference should be at least 100 nm for OM and at least 40–50 nm for SEM. The study of the deformation area around the indentations using OM showed good visualization of cracks, the opening width of which is greater than its resolution – 250 nm (Figure 2a, b). After applying a load of 1.0 N, it was not possible to determine the presence of cracks around the indentation imprint using OM (Figure 2a). After applying a load of 2.0 N near the studied imprint, it was possible to determine the presence and length of three cracks (Figure 2b, Table 3). It was also found that during visualization, the crack is visible with a large opening. Closer to the crack tip, the opening decreases and becomes invisible in an optical microscope. For this reason, the crack length is incorrectly determined.

When examining the same prints in the SEM, from two (Figure 2a, marked with white arrows) to five cracks around the imprint (Figure 2b, marked with white arrows) were detected. The SEM perfectly visualizes cracks with an opening or a height difference of more than 40–50 nm.

AFM made it possible to identify from six (Figure 2a, marked with yellow arrows) to eight cracks around the indentation imprint (Figure 2b, marked with yellow arrows). The absence of cracks after a load of 1.0 N when visualized with an optical micro-

scope shows a 100 % error compared to AFM, i. e. OM reveals nothing compared to AFM or SEM. The accuracy of determining the crack tip on AFM is due to the surface profile.

You can see how a crack of the second type with vertical opening is visualized using OM, SEM and AFM using the example of crack No. 2 in Figure 2b. Schematically, for comparison, this is shown in Figure 2d. On OM this crack could not be determined. The SEM shows only a part of a crack of length l_1 with a large difference (56–73 nm) in height without opening. AFM showed that the crack has a length of l_1+l_2 and is almost twice as large (Table 2) as compared to the SEM value.

Detection of cracks after 2.0 N is due to the larger width of crack opening compared to AFM and makes it possible to detect only 37 % of all cracks. SEM makes it possible to detect from 33 to 75 % of all cracks, depending on the applied load, i. e. the higher the load, the larger the cracks and better visible in the SEM.

Now let's compare the cracks, the length of which was determined by all three methods, as well as the error obtained by incorrect determination of the crack length (Table 3). These cracks correspond to numbers No. 1, 3 and 4 in Table 2 after a load of 2.0 N. As a result, the use of AFM makes it possible to increase the determination accuracy up to 100 % (Table 4).

Table 3

Crack length around indentation imprint and length errors compared to atomic force microscopy

Type	OM				SEM				AFM		
	Load, N	1.0	%	2.0	%	1.0	%	2.0	%	1.0	2.0
Crack No, μm	1			14.01	30	15.80	3	16.40	18	16.29	20.09
	2			–	100	6.30	9	6.94	39	6.39	11.39
	3			10.53	46	–	100	19.80	1	6.78	19.64
	4	no cracks found	100	10.08	24	–	100	10.28	23	4.52	13.31
	5			–	100	–	100	16.50	15	5.97	19.41
	6			–	100	–	100	13.04	0	10.06	13.02
	7			–	100	–	–	–	100	–	10.79
	8			–	100	–	–	–	100	–	9.57

Table 4

Resolution and errors of visualization methods

Type	Resolution, nm	Error in determining, %	
		d	c
OM	at least 250	up to 9.0	up to 100
SEM	at least 1.0	up to 2.3	up to 67.0
AFM	at least 0.2 (by XY), at least 0.03 (by Z)	up to 2.0	

Comparison of methods for determining microhardness

The results of determining the microhardness are shown in Table 5. The values of the elasticity modulus E of the samples were measured on the NI.

After determining a , l , c , E and H , one of the formulas (4) and (5) was selected with respect to c/a . Then K_{IC} was calculated. It has been established that the values of microhardness with NI compared with Vickers differ by 1.2–1.6 times or by 25–38 % (Table 5). This leads to an error in determining the K_{IC} of 16–23 % (Table 5).

Table 5

Values of microhardness, fracture toughness and errors of their determination by two methods

Sample	Microhardness H , GPa			K_{IC} , $\text{MPa}\cdot\text{m}^{1/2}$		
	Vickers	NI	%	Vickers	NI	%
Si (100)	8.6±0.9	13.8±0.6	37.7	0.97±0.05	1.20±0.05	19.2
Si (110)	8.8±0.4	13.6±0.7	35.3	1.00±0.05	1.26±0.09	20.6
Si (111)	8.4±0.3	13.4±0.7	37.6	0.99±0.06	1.19±0.10	16.8
Quartz MRF	10.0±0.7	13.5±0.1	25.9	1.17±0.17	1.40±0.21	16.4
Glass slide	10.1±0.6	6.7±0.1	33.6	1.82±0.04	1.42±0.03	22.0
Cover glass	10.1±0.2	6.4±2.4	36.6	1.45±0.07	1.10±0.05	23.9

Conclusion

Three ways to improve the accuracy of determining of the critical stress intensity factor K_{IC} , which quantitatively characterizes the fracture toughness, were considered: the choice of a mathematical calculation model, the use of atomic force microscopy to visualize the deformation region, and the nanoindentation method to determine the microhardness and elasticity modulus of the material.

It was established that changing the physical principle of the visualization method for cracks and indentation parameters from optical to micro-

mechanical (atomic force microscopy) leads to a decrease of the error in determining of the indent diagonal length by 2.3–9.0 %. This also leads to a decrease of the error in determining of the crack length by 46–100 % compared to optical microscopy and 24–67 % compared to scanning electron microscopy. The method of atomic force microscopy proposed in this work for visualizing of the deformation region due to spatial three-dimensional visualization, atomic forces and high accuracy ($XY \pm 0.2$ nm, $Z \pm 0.03$ nm) can significantly expand the possibilities of using the indentation method. It becomes possible to determine the fracture toughness

of individual phases in a material and individual elements of microelectromechanical systems through the use of small loads.

The use of the nanoindentation method instead of the Vickers method made it possible to increase the accuracy of determining of the material's microhardness up to 38 %, as well as the critical stress intensity factor K_{IC} up to 23 %.

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