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Determination of Crack Resistance of the Cover and Slide Glass by Indentation Method with the Visualization Using Atomic Force Microscopy

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Abstract

Crack resistance of two types of glass was studied – cover glass (0.17 mm thick) and slide glass (2 mm thick) using an improved technique through the use of the probe methods, which makes it possible to increase the accuracy of determining the crack resistance of glass. Colorless silicate glass was used. Crack resistance was determined by the Vickers pyramid indentation method. Microstructure of glasses surface and deformation region after indentation were studied using an atomic force microscope. Mechanical properties of glasses were determined by nanoindentation. Surface relief of a glass slide is rougher than that one of a cover glass. Roughness R_z for a cover glass is less than for a slide glass. Specific surface energy value of 0.26 N/m is higher for the slide glass compared to the coverslip. One elastic modulus value E of the cover glass is 48 GPa, and that one of the slide glass is 58 GPa. The microhardness value H is almost the same for by the glasses and amounts to 6.7 GPa for a slide glass and 6.4 GPa for a cover glass. Atomic force microscope images of deformation region after indentation with a Vickers pyramid show that the first cracks appear at a load of 1 N on the slide glass, and at 2 N on the cover glass. At a load of 3 N, the cover glass is destroyed. Based on the results of crack resistance calculations it was found that critical stress intensity coefficient K_{IC} values are 1.42 MPa·m$^{1/2}$ for a glass slide, and 1.10 MPa·m$^{1/2}$ for a cover glass.

Keywords: cover glass, slide glass, crack resistance, indentation method, atomic force microscopy

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Определение трещиностойкости покровного и предметного стекла методом индентирования с визуализацией методом атомно-силовой микроскопии

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Проведены исследования трещиностойкости стекла двух видов – покровного (толщина 0,17 мм) и предметного (толщина 2 мм) по усовершенствованной методике за счёт применения зондовых методов, позволяющих повысить точность определения трещиностойкости стекла. Использовали бесцветное силикатное стекло. Трещиностойкость определяли методом индентирования пирамидой Виккерса. Микроструктуру поверхности стекла и область деформации после индентирования исследовали на атомно-силовом микроскопе. Механические свойства стекла определяли методом наноиндентирования. Рельеф поверхности у предметного стекла более шероховаты по сравнению с покровным стеклом. Шероховатость R_z у покровного стекла меньше по сравнению с предметным. Удельная поверхностная энергия 0,26 Н/м выше у предметного стекла по сравнению с покровным. Модуль упругости E у покровного стекла составляет 48 ГПа, у предметного – 58 ГПа. Микротвёрдость практически совпадает и составляет 6,7 ГПа у предметного стекла и 6,4 ГПа у покровного. На АСМ изображениях области деформации после индентирования пирамидой Виккерса установлено, что первые трещины появляются при нагрузке 1 Н на предметном стекле, а на покровном – при 2 Н. При нагрузке 3 Н покровное стекло разрушается. По результатам расчёта трещиностойкости установлено, что критический коэффициент интенсивности напряжений K_{IC} для предметного стекла равен 1.42 МПа·м$^{1/2}$, а K_{IC} для покровного стекла равен 1.10 МПа·м$^{1/2}$.

Ключевые слова: покровное стекло, предметное стекло, трещиностойкость, метод индентирования, атомно-силовая микроскопия

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Introduction

Glass is a material which is widely used in everyday life, various technologies, medicine, and microelectronics. However, the glass is a very fragile material. Mechanical properties (including the crack resistance) of the glass may change depending on its composition [1–5]. Internal stresses [6] and glass thickness of the product can also affect to the crack resistance. Glass deformation under load occurs in different ways: from the crack's formation (in which the integrity of the glass as a product is still preserved) to the complete destruction (this is the destruction of the product into parts) [7–9]. The most common method for determining crack resistance is the indentation method [2, 3, 5, 10]. The crack resistance varies from 0.38 to 1.79 MPa·m$^{1/2}$ depending on the composition of the glass [2, 10]. Previously, in [11–13], an improved method for determining crack resistance by the indentation method was described through the use of high-precision probe methods (atomic force microscopy and nanoindentation). This makes it possible to increase the accuracy of determining the crack resistance of brittle solid materials.

The aim of the work was to determine the crack resistance of two types of glass (cover glass and slide glass) using the indentation method with visualization of the deformation area using atomic force microscopy and determination of the mechanical properties by nanoindentation.

Experimental details

Slide glass and cover glass were used for the study. Slide glass type is: colorless silicate glass (according to TU 9464-012-52876859-2014), dimensions 26×26±1.0 mm, thickness 2±0.2 mm. Cover glass type is: colorless silicate glass (according to TU 9464-012-52876859-2014), dimensions 18×18 ± 1.0 mm, glass thickness 0.17 ± 0.02 mm.

The determination of the crack resistance was conducted using the indentation method [11–14]. Indentation imprints were performed on a PMT-3 microhardness tester (LOMO, St. Petersburg, Russia). A Vickers tip was used as an indenter. The load on the indenter varied from 0.01 to 5.0 N. Three indentations were performed at each load.

Structure of the glass surface, topography of the indentation imprints and formed cracks on the glass were determined using a Dimension FastScan atomic force microscope (AFM) (Bruker, USA) in PeakForce QNM mode. The standard silicon cantilevers of the NSC-11 type (Mikromasch, Tallinn, Estonia) were used. The radius of the probe tip is 25 nm and the console stiffness is 6.1 N/m.

Microhardness H, elastic modulus E and deformation η (plastic and elastic deformation) of the glass were determined using a Hysitron 750 Ubi nanoindenter (NI) (Bruker, USA) [15]. A Berkovich diamond indenter with a curvature radius of 60 nm was used. During nanoindentation, the deformation curves are continuously recorded. As a result, data on the applied load and the corresponding the indentation depth are obtained in the form of the dependence function $F = f(h)$. On each sample, 9 indentations were performed with a constant load of 5 mN. Microhardness and elastic modulus were also determined depending on the depth. In this case, the load varied from 0.2 to 10 mN.

The elastic recovery η_{elast} and plastic deformation η_{plast} were calculated from the shape of the stress-strain curves $F = f(h)$ [16]. The mechanical energy W_{tolal} performed during indentation is only partly spent on the plastic energy W_{plast} [16]. When the applied load is removed, part of the energy (elastic recovery energy W_{elast}) is released. Relation (1) contains information characterizing the plastic properties of the test sample:

$$\eta_{elast} = \frac{W_{elast}}{W_{total}}, \quad (1)$$

where $W_{tolal} = W_{elast} + W_{plast}$.

The plastic component is equal to:

$$\eta_{plast} = (1 - \eta_{elast}) \cdot 100\%. \quad (2)$$

To determine critical stress intensity coefficient K_{IC}, the following formulas were used [11–13]:

$$K_{IC} = 0.035 \left(\frac{l}{a}\right)^{-\frac{1}{2}} \cdot \left(\frac{H}{E\Phi}\right)^{-\frac{2}{5}} \cdot \left(\frac{Ha^{\frac{1}{2}}}{\Phi}\right); \quad (3)$$

$$K_{IC} = 0.129 \left(\frac{c}{a}\right)^{-\frac{3}{2}} \cdot \left(\frac{H}{E\Phi}\right)^{-\frac{2}{5}} \cdot \left(\frac{Ha^{\frac{1}{2}}}{\Phi}\right), \quad (4)$$

where P is the load on the indenter, N; l is the crack length near the indentation imprint, m (Figure 1); c is the crack length from the center of the indentation imprint, m; a is the length semi-diagonal of the indentation imprint, m (Figure 1); H is the microhardness, GPa; E is the elastic modulus, GPa; Φ is a constant, an indicator of the bond reaction in the crystal lattice, Φ ≈ 3. The c/a ratio must be determined to select the formula. If $c/a \leq 2.5$, then

Palmquist cracks (Figure 1) form in the sample and the calculation is carried out according to formula (3) [12, 17, 18]; if $c/a > 2.5$, then median cracks (Figure 1) form in the sample and the calculation is carried out using formula (4) [12, 17,18]. Formulas (3) and (4), as already shown in [12], were chosen for calculation as the most reliable. The values of H and E obtained by the NI method were used as microhardness and elastic modulus in K_{IC} calculations.

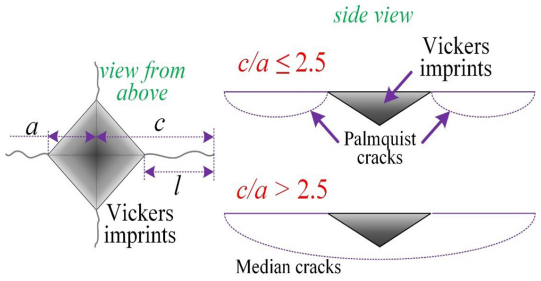

Figure 1 – Indentation imprint with indication of cracks and them type

Results and discussion

AFM-images with the surface profiles of the cover and slide glass are shown in Figure 2. On the surface of the cover glass and slide glass there are stripes oriented in the same direction. The surface relief of the slide is slightly more developed than that of the cover glass (Figure 2*b*). The surface roughness of the slide glass and cover glass they are close in terms of values (Table 1). Only the roughness R_z of the cover glass is lower compared to the slide glass. Specific surface energy for the slide glass (0.26 N/m) is higher compared to the cover glass (0.08 N/m).

Table 1

Roughness (R_a, R_q, R_z) and specific surface energy γ of glass

Glass type	R_a, nm	R_q, nm	R_z, nm	γ, N/m
Slide glass	1.24 ± 0.06	1.58 ± 0.08	3.51 ± 0.18	0.08 ± 0.01
Cover glass	1.06 ± 0.05	1.57 ± 0.08	2.64 ± 0.13	0.26 ± 0.01

Mechanical properties of two types of glass were studied under constant (Table 2) and increasing load (Figure 3) by the nanoindentation method.

Table 2

Mechanical properties of glass

Glass type	E, GPa	H, GPa	η_{plast}, %	η_{elast}, %
Slide glass	58±3	6.7±0.2	37.8±5.7	62.2±9.3
Cover glass	48±1	6.4±0.2	33.7±5.1	66.3±9.9

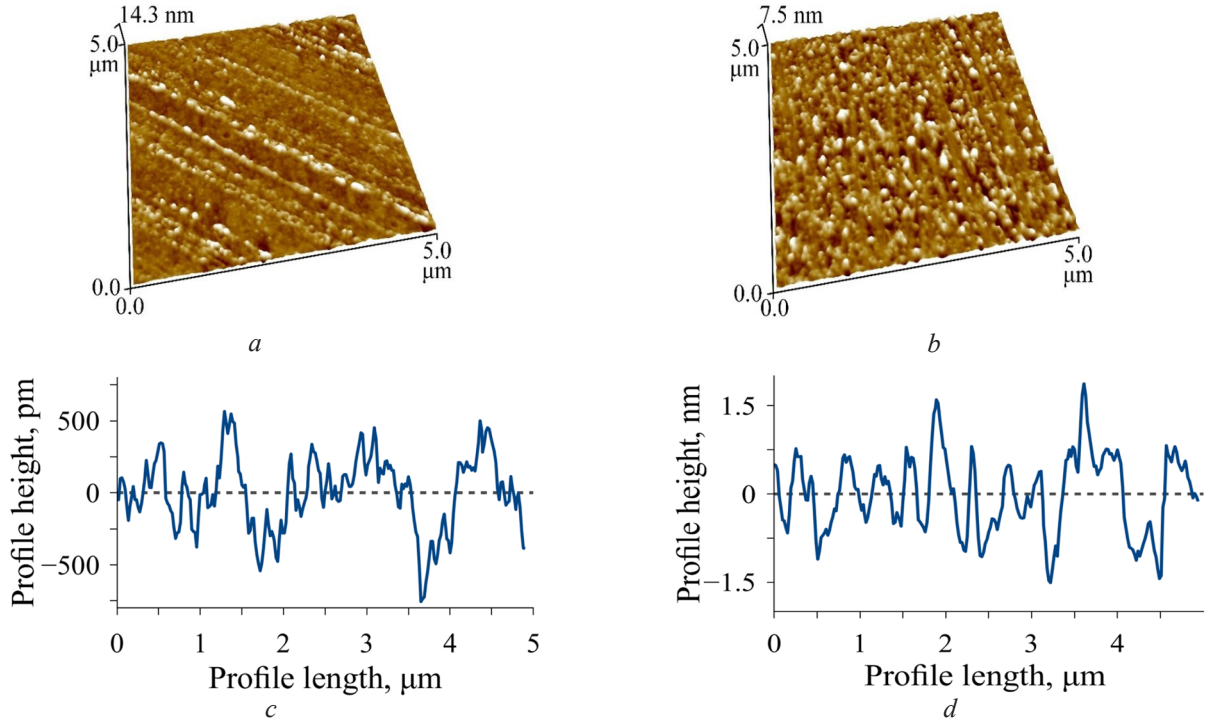

Figure 2 – Atomic force microscope images of the cover glass (*a*) and slide glass (*b*) (in a field of 5×5 μm^2) with surface profiles (*c*, *d*)

Based on the study results of the mechanical properties of glasses at a load of 5 mN (Table 2), it was found that the elastic modulus E of the cover glass is 48±1 GPa, and that of the slide glass is 58±3 GPa. Microhardness H is almost the same and amounts to 6.7±0.2 GPa for the slide glass and 6.4±0.2 GPa for the cover glass. Mechanical properties values are lower than those of pristine silica glass and fully densified silica glass [19]. Microhardness practically coincides with the values in [20]. It can be said that the composition of the glass can significantly affect the mechanical properties and, subsequently, the strength characteristics of the glass.

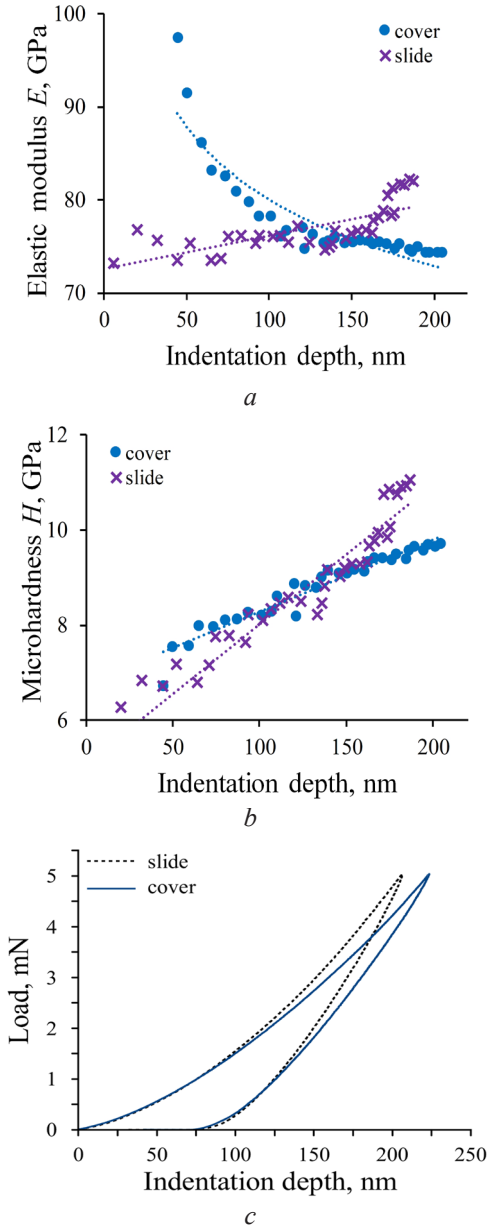

Figure 3 – Dependences of elastic modulus (*a*) and microhardness (*b*) on indentation depth and indentation curves (*c*) on the slide glass and cover glass

When studying microhardness H with increasing load (from 0.2 to 10 mN), it was found that the values are almost the same for both glasses (Figure 3*b*). Elastic modulus E of the slide glass at small depths (up to 70 nm) practically does not change, and then increases to 80–85 GPa (Figure 3*a*). For the cover glass, the elastic modulus decreases with increasing depth from 50 to 100 nm. After depth 100 nm, the elastic modulus remains virtually unchanged – 73–75 GPa. Plastic deformation of the slide glass is higher than that of a cover glass and amounts to 37.8 % and 33.7 %, respectively (Table 2, Figure 3*c*).

Morphology of the indentation imprints on the glasses surface has been studied after studying the surface morphology of the glasses and mechanical properties. It has been established first cracks appear at a load of 1 N on the slide glass, and on a cover glass – at 2 N using the AFM method. Cracks (Figure 4), which appear parallel to the edges of the imprint at low loads (up to 1 N), are breaks, not cracks [2]. These breaks initiate the appearance of the radial cracks when large loads are applied [2]. The structure of the deformation area is similar to the structure in [2, 7, 21, 22]: there are imprints on the glass surface with breaks along the verges of the imprint and cracks spreading from the corners of the imprint.

Also, from the AFM images, the diagonals length d and the indentations depth h for the slide and cover glasses were determined (Figure 5). For both the slide and cover glass, the values of h and d are almost the same up to a load of 2 N. Cover glass is destroyed at a load of 3 N (Figure 5*b*). The c/a ratio for the cover and slide glasses is given in Table 3.

Table 3

c/a ratio for slide glass and cover glass

Load, N	c/a	
	Slide glass	Cover glass
1	1.3 ± 0.1	–
2	2.0 ± 0.2	2.2 ± 0.2
3	2.1 ± 0.2	destruction
5	2.2 ± 0.2	destruction

It is established that $c/a \leq 2.5$ and Palmquist cracks form in the glasses (Table 3). The critical fracture intensity coefficient K_{IC} should be calculated using formula (3).

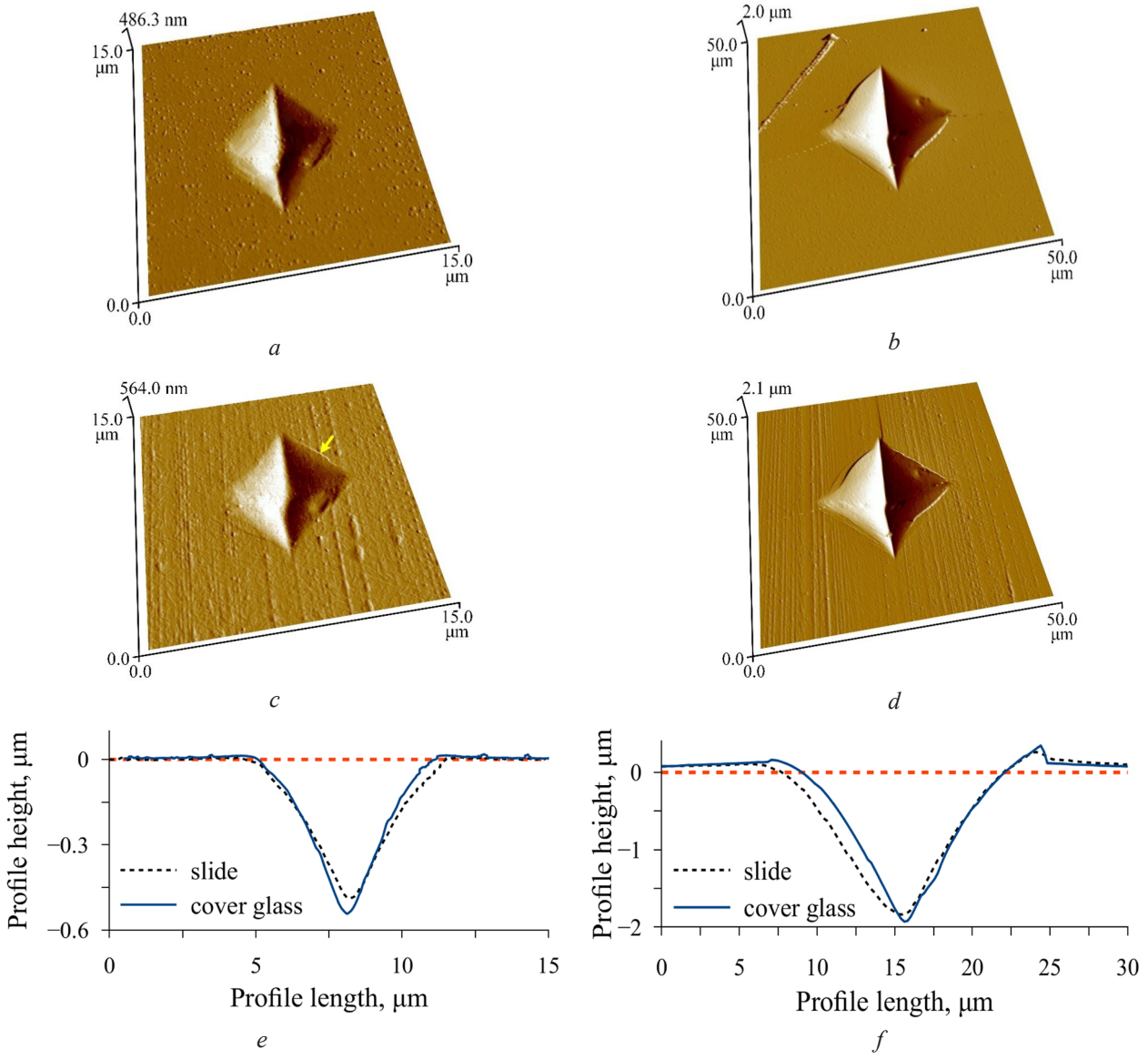

Figure 4 – Atomic force microscope images of the indentation imprints on the surface of a cover glass (*a*, *b*) and slide glass (*c*, *d*) at 0.01 N (*a*, *c*, *e*) and 2 N (*b*, *d*, *f*) and their profiles

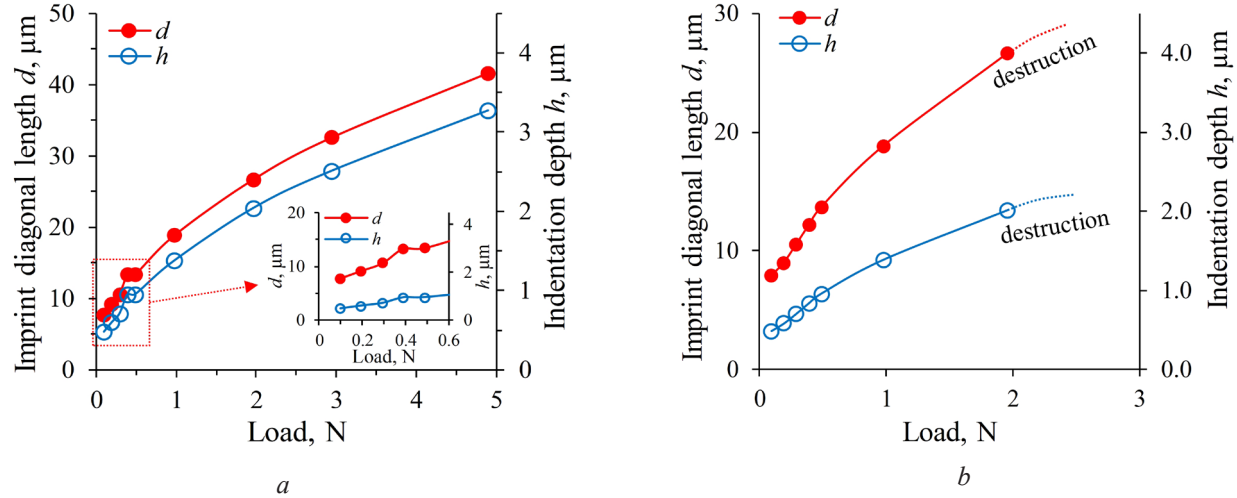

Figure 5 – Dependence of the diagonal length and indentation depth on the load for slide glass (*a*) and cover glass (*b*)

According to the calculation results, it was found that K_{IC} for the slide glass is equal to 1.42±0.03 MPa·m$^{1/2}$, and K_{IC} for a cover glass is 1.10±0.05 MPa·m$^{1/2}$. The K_{IC} values obtained in this work are close to the values in [10] for silicon oxynitride glasses and B_2O_3-based glasses.

The destruction of the cover glass under a load of 3 N is associated with its smaller thickness compared to the slide glass. Thus, the glass thickness can influence the value of the fracture toughness: the thinner the glass, the lower its breaking force.

Conclusion

The crack resistance of two types of glass (slide glass and cover glass) was determined using an improved indentation method. The used of probe methods (atomic force microscopy and nanoindentation), which allowed it possible to accuracy increase of determining the crack resistance of glass. Colorless silicate glass was used.

Using a Vickers pyramid was carried out indentation. The deformation area was visualized using atomic force microscopy. The mechanical properties of glasses were determined by nanoindentation (at a constant load and with increasing load).

Based on atomic force microscopy images, it was established that there are stripes oriented in the same direction on the surface of the cover glass and slide glass. The surface relief of a slide glass is slightly more developed than that of a cover glass. The specific surface energy is higher for the slide glass (0.26 N/m) compared to the cover glass (0.08 N/m). The elastic modulus E of the cover glass is 48 GPa, and that of the slide glass is 58 GPa. The microhardness H is almost the same and is 6.7 GPa for the slide glass and 6.4 GPa for the cover glass. The elastic modulus E of the slide glass at small depths (up to 70 nm) practically does not change, and then it increases to 80–85 GPa. For a cover glass, the elastic modulus decreases with increasing depth up to 100 nm, and then remains virtually unchanged (73–75 GPa).

Atomic force microscopy images of the deformation area after indentation with a Vickers pyramid show that the first cracks appear at a load of 1 N on the slide glass, and at 2 N – on the cover glass. The cover glass is destroyed at a load of 3 N. The diagonals length d and the indentations depth h for the slide and cover glasses are practically the same up to a load of 2 N. It was found that Palmquist cracks form on glass of both types during indentation. The crack resistance K_{IC} for a slide glass is 1.42 MPa·m$^{1/2}$, and for a cover glass is 1.10 MPa·m$^{1/2}$.

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