Вплив поверхневих і внутрішніх дефектів на руйнування та конструкційну міцність скляних матеріалів / Родічев Ю., Трегубов Н., Веер Ф., Маслов В. // Вісник ТНТУ. — 2011. — Спецвипуск — частина 1. — С.114-123. — (механіка та матеріалознавство).

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ВПЛИВ ПОВЕРХНЕВИХ І ВНУТРІШНІХ ДЕФЕКТІВ НА РУЙНУВАННЯ ТА КОНСТРУКЦІЙНУ МІЦНІСТЬ СКЛЯНИХ МАТЕРІАЛІВ

***Резюме**. Експериментально досліджено особливості крихкого руйнування та конструкційної міцності скляних матеріалів з поверхневими, внутрішніми та змішаними типами критичних дефектів при короткочасному і довготривалому механічному навантаженні. Флоат скло і багато композитних скломатеріалів є поверхнево дефектними структурами з мікророзмірними поверхневими тріщинами та пошкодженнями, стан яких визначає опір руйнуванню і працездатність силових конструкцій. Показано, що характерні типи та розміри критичних дефектів склокерамічних структур можуть змінюватися суттєво залежно від технологічних особливостей застосованої „скляної” або „керамічної” технології виробництва. Механічна поведінка силових конструкцій зі скла і склокераміки може бути ефективно керована за рахунок цілеспрямованого вдосконалення технології і запровадження методів контролю критичних дефектів та моніторингу міцності на поточному виробництві. Результати дослідження і позитивний досвід проектування міцних пластин, балок та оболонок дозволяють надійно гарантувати несучу спроможність навантажених конструкцій з різних видів скломатеріалів.*

***Ключові слова:** скломатеріали, поверхневі мікротріщини, внутрішні дефекти, крихке руйнування, конструкційна міцність.*

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INFLUENCE OF SURFACE AND INTERNAL DEFECTS ON THE FRACTURE AND ENGINEERING STRENGTH OF GLASS MATERIALS

***The summary.** Features of fracture and engineering strength of glass materials with surface and internal and mixed modes of critical defects are studied experimentally under short term and long term mechanical loading. The float glass and many composite glass materials are surface defective structures with micro dimensional surface cracks and damages. State of these defects determines the strength and workability of carrying structures. It is stated that characteristic modes and sizes of critical defects of glass-ceramic structure may be changed significantly subject to technological peculiarities of concrete “glass” or “ceramic” production method. Mechanical behavior of carrying glass and glass-ceramic structures may be directed using the appropriate technology, methods of critical defects control and strength monitoring in the production line. The results of the study and positive experience of the design of strong plates, beams and shells show the possibility to guarantee fully the carrying capacity of load bearing constructions made of different kinds of glass materials.*

***Key words:** glass materials, surface micro-cracks, internal defects, fracture, engineering strength.*

Symbols

LMF – linear fracture mechanics;
σ_{1cr} – maximal value of the bending stress, MPa;

σ_d - long term strength of glass, MPa;

K_{Icr} – critical value of strength intensity factor K_I, MPa√m;
b_0 – initial depth of the micro-crack in the fracture focus, m;
b_1 – critical depth of the micro-crack in the fracture focus, m;
A_1 – length of the critical micro-crack in the fracture focus, m;
Y geometrical factor is calculated basing on linear fracture mechanics, experimental value b_1/ $0.5A_1$, test data on the orientation and shape of surface micro-crack in the fracture focus;
t_d – lifetime of the parts under durable static loading with constant stress level σ_d, s;
V_b – speed of micro-crack moving, m/s;

Introduction. Glass and ceramics are specific brittle structural materials with an uncertain level of strength. The main difference from conventional structural materials is a lack of ductility and an extremely small size of defects such as micro-cracks, causing brittle fracture [1, 2]. The control of critical defects and actual strength parameters for glass and ceramics is a special sphere of brittle material science which is not developed enough up to now. For these reasons, the engineering strength and durability of building and automotive glass and technical ceramics are not normalized as reliably as for metal and other traditional structural materials. Glass producers give not any guarantee of actual strength of their products.

The problem can be solved using the experience of creating durable products for special purposes - aviation glass, optoelectronic devices, aerospace and deepwater technology [1-4]. The latest studies of the engineering strength of glass and ceramics in relation to building structures showed promising ways of increasing performance of bearing elements made of these materials [5-7]. However, the complex of knowledge in this area remains little known to a wide range of specialists in architectural and automotive glass up to now. This reduces the efficiency of glass and ceramics use as a structural material.

Engineering strength of this class of materials depends on a variety of technological, structural and operational factors [3, 4]. There is presented an integrated technical approach to create load-bearing elements of glass and glass-ceramics with a given level of strength and durability. The management of glass-material strength by controlling defects and optimization of production technology on strength criteria is the basic part of this approach. This approach allows to consider the impact of the main technological, constructional and operation factors on the actual state of critical defects and carrying capacity of structural elements.

The paper deals with specific surface and internal defects in the glass and ceramics. It is shown the predominant influence of surface defects on the strength of homogeneous amorphous materials such as glass with nanoscale defects in the internal structure. It is found an increase in the role of technological internal defects in the glass-crystalline materials obtained by using different technological methods. The higher are heterogeneity and defectiveness of the structure made of glass material the stronger is the influence of internal defects. The result - a mixed mode of fracture and the transition to predominantly internal fracture sources in very inhomogeneous and defective ceramic products made by slip ceramic technology.

It is shown that mechanical behavior of carrying glass and glass-ceramic structures may be directed using the appropriate methods of critical defects control, strength monitoring in the production line as well as new technologies optimized on the strength criteria. The results of the study and positive experience of the design of strong plates, beams and shells show the possibility to guarantee fully the carrying capacity of load bearing constructions made of glass and glass-ceramics.

Defects and strength of surface defective homogeneous materials. Float glass and high quality heat-resistant glass ceramics produced on glass technology are the surface defective materials. It was shown that the surface micro-cracks placed in the specific surface cracked layer are the main fracture sources under mechanical and thermal loading of carrying parts [1, 6]. Thanks to high quality and homogeneity of structure, the internal defects are rarely lead to the destruction of structural elements. The typical semi-elliptical micro-crack can be found usually in the center of the mirror zone on the fracture surface of the elements tested under the bending or tension as shown in Figure 1.

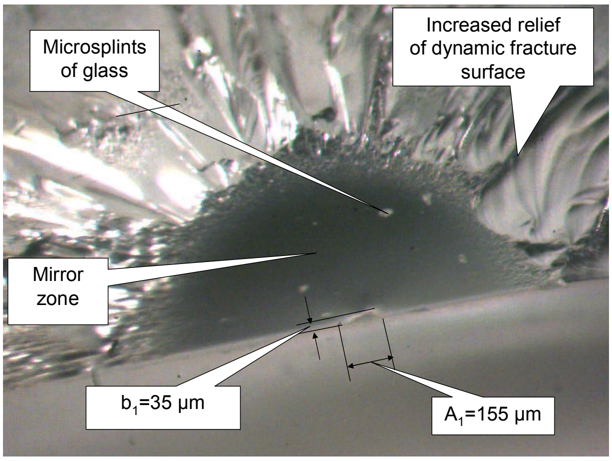


Figure 1. Micro-crack in the center of mirror zone on the fracture surface of flat structural element made of the tempered float glass

This type of critical defects is character for “central” mode of fracture sources have formed at some distance from the edge of the details. Critical micro-crack is formed usually in the process of loading at the stress value closed to the maximum magnitude. Deepest and more movable initial micro-crack or set of micro-cracks located in the cracked surface layer are the basis for the formation of crack with critical shape and depth in the fracture focus. Therefore, the strength and durability of parts made of surface defective materials depends directly on the mode and depth of the cracks formed in cracked surface layer under the structural element working.

The depth and other parameters of the cracked surface layer can be controlled with special micrographic search of the fracture surface of tested specimens to guarantee a certain level of strength of float glass. It is not possible to assess the state of cracked surface layer and damages under direct search of glass element surface using the optical microscope. The surface signs of scratches, other defects and damages give not the important information about character and depth of micro-cracks which is need for strength assessment using the fracture mechanics approach (Figure 2).

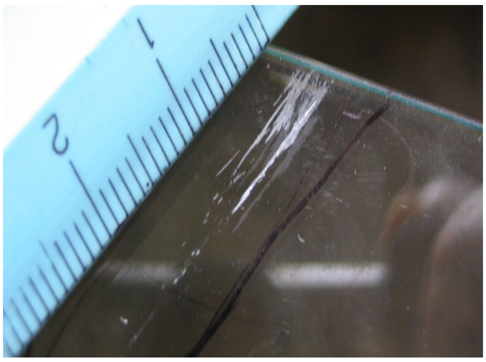


Figure 2. Set of scratches on the surface of the glass plate after the tempering. Subsurface parts of cracks are not seen.

It was found that under special bending tests some important information about the micro-thin cracked surface layer may be discovered on fracture surface in “compressed” side of plates. The method of cracked layer control for elements made of float glass was developed basing on this effect. The Figure 3 shows the cracked surface layer with a thickness 30… 45 μm detected by a special control breakage of glass part. The test results can be used to select the best type of glass and the manufacturer, as well as to control the quality of structural element processing.

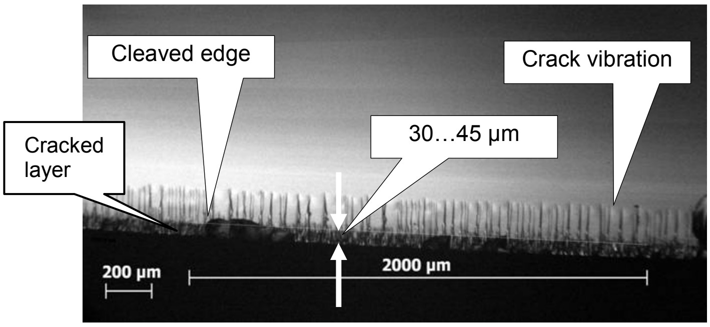


Figure 3. Results of quality control of float glass by assessment of structure and depth of the cracked layer

Using the new technique, the subcritical growth of the long surface crack in glass plate under the bending test was found at a load close to the limit. The Figure 4 shows that the subcritical growth of crack reaches 84% of its initial depth of crack. The problem is to choose the basic geometrical parameter – initial depth b_0 or critical depth b_{cr} for the assessment of the life time and crack resistance of glass currying elements using the equations of fracture mechanics [6]. The critical dimensions of the micro-crack correspond to transient final phase of glass destruction. Their control is more difficult than the initial parameters of the crack. Therefore, the initial crack depth was accepted in estimates of crack growth resistance and strength of surface defective materials.

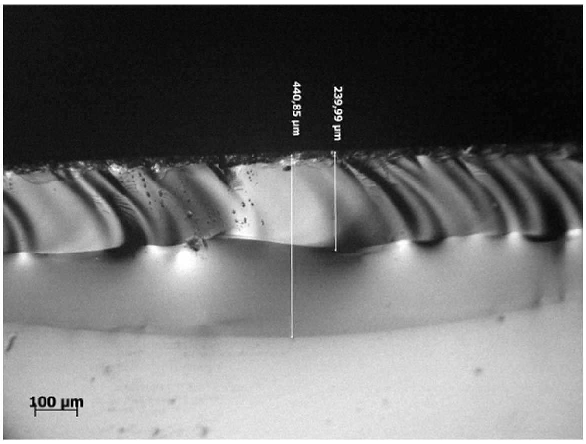


Figure 4. Subcritical growth of the long surface crack in glass plate
at the approximately 95% of the failure bending stress

The almost ideal linear elastic behaviour of glass is observed on both macroscopic and microscopic levels. Therefore, the theory of linear facture mechanics (LFM) may be used to assess the micro-fracture parameters, structural glass strength and durability in tension and bending. Next method of micro-crack inspection and glass strength assessment was used for parts studied under short-term loading.

The values for the bending strength closely approximate to experimental data on short time glass strength may be calculated using the equation:

$$\sigma_{Icr} = K_{I\,cr} / Y\sqrt{b_1} \tag{1}$$

Some basic data on critical micro-cracks were discovered:

- Semi-elliptical and long surface micro-cracks are usually the source of a "central" fracture;
- The quarter elliptical or quarter circular geometries are typical for edge micro-cracks;
- The counter of critical crack at the stage 1 of its stable growth is a curve connecting with the surface of "mirror zone";
- Micro-crack growth is unstable on the next stages, when crack counter moved on the surface of "mirror zone" and further;
- The experimental values of the parameter Y_I calculated using the micrographic data and LFM equations are in the range of 1.36 to 2.0.

• The statistical heterogeneity of the data on factor *KIcr* , parameter *Y1* and *b1* causes the significant scatter of the engineering strength of glass.

• Only by using micrographic data can statistical distributions be evaluated properly.

Time dependencies of strength and fracture of glass.

Time dependencies of strength and fracture characteristics of glass may be controlled using the experimental kinetic $K_I - V_b$ diagrams (Figure 5) for defined types of glass under fixed operational conditions.

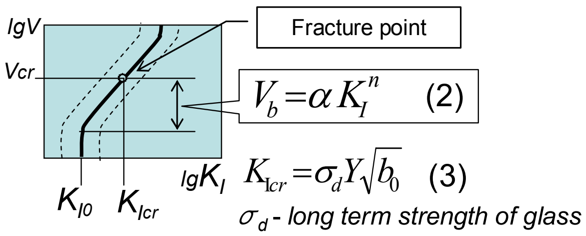


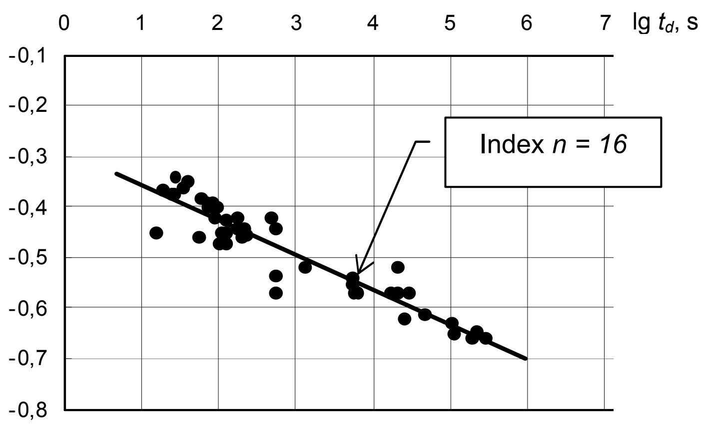
Figure 5. Experimental $K_I - V_b$ diagrams and subcritical growth of the surface crack in glass

Subcritical stable slow accelerated crack growth in glass under constant value long-term stress σ_d occurs at the factor K_I changing from initial value K_0 to critical value K_{Icr}. The actual problem is to assess the values of the factors α and n in the equation (2) for different conditions of glass surface, working environment and modes of element loading.

Minimal V_b for sub-critical stable crack growth in glass under static bending was in the range 10^{-10} m/s to 10^{-8} m/s , as maximum V_{bcr} was 10^{-4} to 10^{-3} m/s. Values of factors α = 0.6 m/s and n = 16 were used in some published works.

The actual influence of technological and operational factors on these parameters values is not known.

Figure 6 shows the decrease of critical value of the factor *KIcr* at the tests of cut flat specimens of glass 4 mm in thickness (depth *b0* =350μm, *Y1* = 1.8) in durable bending at constant load.



lg K_{Icr},
MPa√m

Figure 6. Experimental curve for fracture toughness of float glass in durable bending

Critical values of the factor K_{Icr} , critical crack growth speed V_{bcr} , as well as factors α and n are not constant. They can change significantly depending on loading speed, time of durable loading, service conditions and many other factors. These parameters must be controlled.

The equation (4) may be used for practical assessment of life time basing on results of control of the initial crack depth b_0 like this:

$$t_d * = \left[(0,5n-1)\alpha\sigma_d^n Y^n b_0^{\ 0.5n-1}\right]^{-1} \quad (4)$$

The specimens of 6 mm float glass with the surface micro-crack-cuts (b_0 = 310 $\pm$ 10 µm) were tested in bending. Short time σ was 20.5MPa. Mean t_d = 420s was received in long duration tests under σ_d = 16.5 MPa. Values of σ_d for lifetimes t_{dl} - 10s and 10^8s calculated with equation (4) for n =16 and 12 are given in the table.

n	t_d= 10s	t_d = 420s	t_d=10^8s
16	20.8 MPa	16.5 MPa	**7.6 MPa**
12	22.5 MPa	16.5 MPa	**6.0 MPa**

Very low values of long-term strength are the reason of low carrying capacity of structural elements made of annealed glass. The technologies of glass strengthening and surface protection against damage are required to increase the workability of surface defected brittle materials.

It was shown in the study that control of the surface defectiveness, strength and fracture behavior of glass and high quality heat-resistant glass ceramics produced on glass technology is required at all stages of production of load bearing structures as given in Figure 7.

This system of production control includes a set of tests methods – optical control of surface defects and fracture surface of test specimens, mechanical tests of the strength and crack growth resistance under the bending, assessment of strength and life time of the carrying structures basing on specimen test results, as well as on the test results of real structures.

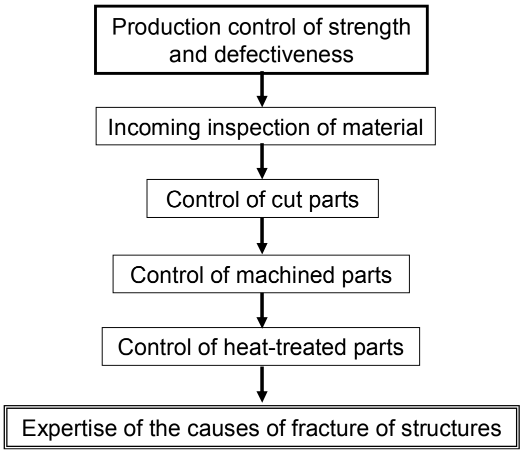


Figure 7. Scheme of production control of strength and defectiveness for glass structural parts

The influence of high localized fracture mode of homogeneous brittle materials, role of scale effect, technological and constructional factors, as well as of usual and extreme operation conditions may be accounting at the implementation of this approach. The number and sequence of testing stages of glass and glass-ceramics ware depends on the method and main stages of manufacturing.

Defects and strength of slip cast glass-ceramics. Considered approach and methods to control the strength and defects of glass products need to be improved taking into account the specifics of the internal and surface structure of glass-ceramics. The mechanical strength of glass-ceramics parts greatly depends on micro-defects of surface and internal structures substantially connected with the factual quality of used production technology, mechanical and heat treatment [8, 9].

It is known that replacing microscopic particles by nano-particles in a composite formulation results in improved operating ability. The authors investigated the strength and fracture of the slip cast heat-resistant glass-ceramics comprising the additives of nano-particles SiO_2 and TiO_2 in the slurry.

Next shortcomings are typical for water slurries based on lithium-silica-alumina glass:

- high humidity of the suspensions is need to guarantee the required fluidity;
- tendency to sedimentation and inhomogeneous densification;
- insufficient density and strength of cast products.

Thermoresistant nano-filled glass-ceramics was developed using the slip ceramic technology and nano-sized additives to improve the internal and surface structure of the material and to increase the strength of shells for extreme operation conditions. The authors investigated the possibility of the adding of nanoparticles SiO_2 and TiO_2 in an amount of 10-30% in the slurry to produce the advanced glass-ceramics ware.

Special set of non-destructive and destructive test methods [7-10] was significantly developed to ensure the durability of glass-ceramics structures in usual and extreme service conditions with the temperature up to 1000 ˚C. Methods based on precise tests of control specimens, samples and real structural elements such as shells and plates were supplemented with other non-standard testing methods for effective use in the operating conditions typical for the production lines of super heat-resistant glass-ceramic.

It was shown in this study that significant heterogeneity, porosity and technological defects of the sintered blanks decrease the strength and carrying capacity of the glass-ceramics shells under normal and extreme conditions. The characteristics of nano- and micro-dimensional structure of raw glass and sintered glass-ceramics were studied using the micrographs made with atomic-force microscope. It was shown that homogeneous structure of special raw glass is close to amorphous structure of float glass (Figure 8). But primary crystallization centers were found in the fracture surface of the special glass blank before its thermal working to realize the process of crystallization.

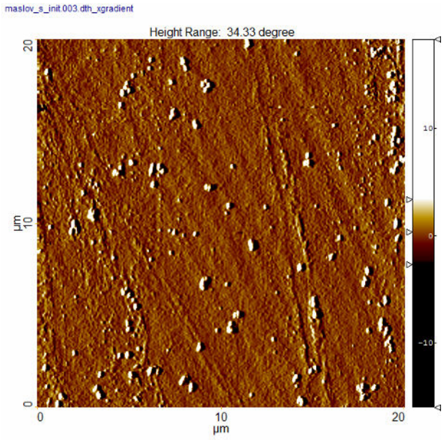


Figure 8. Structure of fracture surface of raw special glass with primary crystallization centers and linear marks of crack discontinuous motion

The character, large scale elements and defects of the internal structure for sintered glass-ceramics ware are shown in the Figure 9. The grain sizes are about 5…10 µm. The sizes of crystalline aggregates were about 20…30 µm. The pores and other internal technological defects together with micro-dimensional defects of glass-ceramics structures are the typical fracture source.

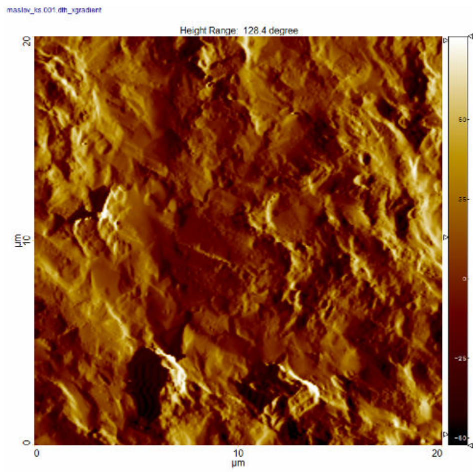
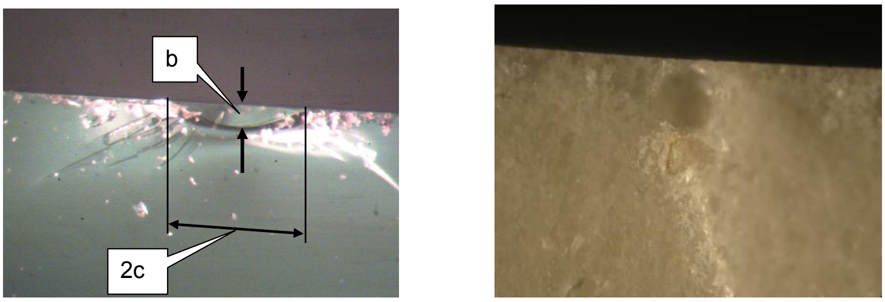


Figure 9. Character of fracture surface of sintered glass-ceramics with enlarged sizes of polycrystalline structure elements and defects

The crack formation and moving take place in the border of glass-ceramics polycrystalline aggregates. The pores and other technological defects may be controlled by special technology, mechanical tests, optical inspection and technological methods. Typical shape and size ratio *b*/2c of the critical microcrack in focus breakage is shown in Figure 10a. The pore and large block of grains in the focus breakage of the slip glass-ceramics is shown in Figure 10b. Scattering damage growth more noticeable effects on fracture behavior and strength of glass-ceramics.



a b

Figure 10. The difference in the nature of fracture of glass and slip glass-ceramics under the bending

New engineering approach was developed to increase the manufacturability and to limit the negative influence of typical shortcomings of the slurry ceramic method. This approach foresees the use of new nano-components in the slurry composition, slip production method and technology of glass-ceramics ware forming. The main idea considered that replacing microscopic particles of SiO_2 by nano-particles in a composite formulation results in improved physical properties.

Technological control of glass-ceramics ware quality and strength included complex assessment the values of bending strength and hardness of the specimens made of real shells, as well as fragments of blanks and shells at the different stages of manufacture production. The results of the study show that adding nano-components limits the sedimentation of coarse fractions slurry, stimulates the suspension homogeneity increasing and compaction of sintered glass-ceramics structure. The bending strength 120 MPa is guaranteed and fracture toughness minimal values increase on 15…20%.The correlation curves for these data were established and used widely in real working environment. The correlation curves for dynamic hardness data and temperature of the glass-ceramics heat treatment are given in the Figure 11. The optimal heat treatment conditions were selected using these experimental regularities for different slurry compositions.

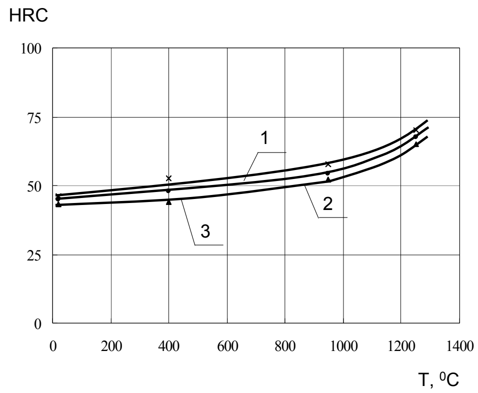


Figure 11. The dynamic hardness data against to temperature of heat treatment of glass-ceramics ware
1 – minimal values; 2 – maximal values; 3 – mean values

As a result of the research found that the adding of nanoparticles in the slurry increases the density of the internal structure and strength of the elements, improves the physical properties and provides high stability of glass-ceramics. It was shown high efficiency of complex engineering approach based on the use of quality and strength test results together with new technological decisions.

The results of this investigation were implemented in the main stages of production of the thermoresistant nano-filled glass-ceramics ware for extreme operation conditions.

Conclusion

From the results it is concluded that:

- the features of fracture and engineering strength of glass materials with surface, internal and mixed modes of critical defects, as well as localized and scattering damage depend on the structure of brittle material due to the influence of technology, constructional and operational factors;
- effective technical approach and methods developed to control the strength and defects of glass products with different modes of defectiveness can be implemented in the main stages of production of the glass and heat-resistant nano-filled glass-ceramics ware for normal and extreme operation conditions;
- depth of the cracked surface layer and the characteristics of microcracks are the important parameters of quality of float glass and processed constructional elements, which is necessary to normalize at the production of bearing glass constructions.

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