SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Study of the stress state of polycarbonate monolithic sheets using optical-polarization methods

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Abstract

Objectives. *The study assessed the possibility of using optical-polarization methods to test quantitatively the stress state and residual stress in polycarbonate (PC) monolithic sheets. This stress is the leading cause of the cracking of PC sheets and the products made of them.*
Methods. *The objects were samples of monolithic PC sheets made by various manufacturers (Monogal and Novattro). The birefringence method was used to study the stress state of the samples, and the interference images obtained in polarized light in crossed polaroids were analyzed.*
Results. *The efficiency of optical-polarization research methods, such as the birefringence and the analysis of the interference images of stretched PC samples combined into an additive spectrum, was shown. The residual stress in the monolithic PC sheets made by various manufacturers was estimated.*
Conclusions. *The quantitative relationship between the stress acting on the PC samples, their birefringence, and the characteristics of their additive spectrum of interference images of stressed*

samples obtained in polarized light in crossed polaroids was established. The possibility of a quantitative assessment of the residual stress in monolithic PC sheets based on an analysis of their additive spectrum of interference images was shown. The measured residual stress did not exceed 1 MPa.

Keywords: *monolithic polycarbonate sheets, stretching, residual stress, crack resistance, birefringence, interference image*

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НАУЧНАЯ СТАТЬЯ

Оценка напряженного состояния поликарбонатных монолитных листов оптико-поляризационными методами

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Аннотация

Цели. *Работа посвящена изучению возможности использования оптико-поляризационных методов для количественной оценки напряженного состояния и остаточных напряжений в поликарбонатных монолитных листах. Эти напряжения являются основными причинами растрескивания листов поликарбоната и изделий из них.*
Методы. *Объектами исследования являлись образцы поликарбонатных монолитных листов различных производителей («Monogal» и «Novattro»). Для исследования напряженного состояния образцов использовали методы двойного лучепреломления и анализ интерференционных изображений образцов, полученных в поляризованном свете в скрещенных поляроидах.*
Результаты. *Показана эффективность использования оптико-поляризационных методов исследования: двойного лучепреломления и анализа характеристик объединенного спектра интерференционных изображений напряженных образцов поликарбоната. Проведена оценка остаточных напряжений в монолитных поликарбонатных листах различных производителей.*
Выводы. *Установлена количественная связь между натяжениями, действующими на образцы поликарбоната, их двойного лучепреломления и характеристиками объединенного спектра интерференционных изображений нагруженных образцов, полученных в поляризованном свете в скрещенных поляроидах. Показана возможность количественной оценки значений остаточных напряжений в монолитных листах поликарбоната на основе анализа их спектров интерференционных изображений. Измеренные остаточные напряжения не превысили 1 МПа.*

Ключевые слова: *листовой монолитный поликарбонат, остаточные напряжения, трещиностойкость, двойное лучепреломление, интерференционное изображение*

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INTRODUCTION

Studying the stress state of organic glasses and their products is necessary for more accurate predictions of their service life [1, 2]. In addition to the external stress that accelerates the cracking of glass during operation, for example, mechanical (upon glazing) or thermal stresses (upon their nonuniform heating), internal residual stress is also "harmful," for example, when it arises in the plexiglass molding process [3]. The following technological conditions for the occurrence of increased residual stress in the manufacture of an extruded monolithic polycarbonate (PC) sheet can be distinguished:

- high viscosity of the PC polymer melts during workpiece molding;
- low temperatures during PC sheet calibration;
- high deformation of the workpiece during PC sheet calibration;
- the high rate of sheet PC calibration;
- the high rate of PC sheet cooling;
- nonuniformity of the PC sheet heating and cooling;
- low-quality of PC sheet cutting;
- incorrect PC sheet storage.

The physical and chemical nature of the influence of external and internal stress on the acceleration of cracking of organic glasses and products made from them is the same; this is a deformation effect on the molecular bonds of the polymer. The cracking process can be considered within the framework of Zhurkov's theory of durability [2, 4]:

$$\tau_p = \tau_0 exp(\frac{U - \nu\sigma}{RT}) \quad (1)$$

where τ_c is the time before the onset of PC cracking (s); U is the activation energy for the destruction of the polymer macromolecules determined by the strength of the PC chemical bonds, equal to 150–160 kJ/(mol·K) [5]; σ is the applied stress (Pa); T is the temperature (K); τ_0 is a constant related to the frequency of vibrations of atoms of molecules upon their thermal motion (τ_0 = 10^{-12}–10^{-14} s); ν is the activation volume of destruction or structure-sensitive parameter (m^3); R (8.314 J/mol·K) is the universal gas constant.

There are methods for estimating the stress state in plexiglasses and their products[1]. These methods are based on the conclusions of Zhurkov's theory (Eq. 1) and are related to the acceleration of plexiglass cracking at elevated temperatures [6] and stresses [1, 2], as well as when their surface comes into contact with adsorption-active test liquids [7, 8]. Several attempts have been made to relate the times of accelerated cracking quantitatively with the residual stress in PC sheets [8]. However, these methods lead to the destruction of the products. Test liquids[2] can only be used for final control of the finished products, indicating the permissible stress level in PC sheets [8].

Nondestructive methods for estimating the stress state in plexiglas can be optical-polarization methods, i.e., the method of measuring birefringence (BR), as well as an analysis of interference images of samples in crossed polaroids (IICP) obtained in polarized light [9, 10]. The essence of the latter method is as follows. BR results in cyclic changes in the intensity of polarized white light transmitted in the material upon loading. This leads to the appearance of an interference image with alternating color bands called isochromes [9–17]. The points on these isochromes correspond to specific values of BR and stress. For uniaxial loading, this stress can be calculated as follows [11]:

$$N = C\sigma\frac{\delta}{\lambda} \quad (2)$$

[1] GOST P 51372-99. State standard of the Russian Federation. *Accelerated life and storable life test methods in special aggresive and other special media for technical products, materials and systems of materials*. M.: Gosstandart; 2000 (in Russ.).

[2] *Bonding and general data on adhesives*. Practice Guide of the compeny EVONIK-RÖHM GmbH; 2011. URL: https://orgsteklo-shop.ru/articles/

where σ is the effective stress (Pa); N is the order (number) of the isochrome bands on the IICP; δ is the thickness (m), λ is the wavelength of light (m); C is the optical sensitivity of the material in terms of stress (1/Pa).

Currently, these techniques are commercially used to assess the quality of PC blanks for laser disks [13]. On the other hand, they can be used more widely, e.g., estimating the residual stress in PC sheets and products made from them [14–20]. This study examined the possibility of using optical-polarization methods to assess the stress state and residual stress quantitatively in monolithic PC sheets.

EXPERIMENTAL

The objects of the study were samples of monolithic colorless light-stabilized 3 mm thick PC sheets, Novattro (*SafPlast*, Kazan, Russia, TU 2246-03-81057157-2008), as well as monolithic colorless light-stabilized 3 mm thick PC sheets, Monogal (*Polygal Vostok*, Kurovskoye, Russia, TU 2246-02-93726592-2008). The test specimens were plates, 100 ± 1 mm long, 3.0 ± 0.1 mm thick, and 30 ± 1 mm wide.

Experiments on the PC sample loading were carried out on an AI-7000-LA5 universal testing machine (Instron) (*GOTECH Testing Machines Inc*., Taiwan). The samples were fastened in the testing machine clamps using a torque wrench [2]. This ensured the constancy and uniformity of pressure on the pulling clamps on the ends of the tested samples. The sample loading was accompanied by continuous fixation of the current elongation (L_τ, m) and load (P_τ, N). The test section length before loading (L_0) was 60 mm. Relative deformation (ε) and stress (σ, MPa) were calculated automatically. The stress σ was calculated considering the decrease in the initial cross-section (s_0, m^2) of the sample upon deformation:

$$\sigma_\tau = P_\tau(1-\varepsilon_\tau)/s_0\,, \tag{3}$$

$$\varepsilon_\tau = (L_\tau - L_0)/L_0\,. \tag{4}$$

A polarizing microscope MIN-10 (*Geologorazvedka*, Russia) with a rotary compensator was used to assess the stress state of the samples by the BR method according to GOST 3519-69[3] [3]. After measuring the compensating rotation angles of this compensator (α and β), the parameter F, which is proportional to the sample BR value (Δn), was then calculated using the following equation:

$$F = d\left(\frac{\alpha-\beta}{2}\right)^e \tag{5}$$

where F is the sample BR parameter (nm); α and β are the angles of the compensator rotation relative to the neutral position (deg); d and e are the device constants (d = 3.085 nm, e = 1.985) [2, 3]. The value of Δn was calculated considering the test sample thickness (δ) using the following formula [3]:

$$\Delta n = F/\delta\,. \tag{6}$$

The test samples were loaded at 20°C until the specified stress was reached [1, 2], and the sample was kept loaded for 600 s until the values of α and β were measured. Measurements were then taken using a polarizing microscope MIN-10.

A digital camera with a microlens adjusted to capture an image at specified time intervals (5 s) was used to fix the IICP of the loaded samples in transmitted polarized light.

RESULTS AND DISCUSSION

The BR of the original unloaded monolithic PC sheets was evaluated first. Previously, transverse strips were cut across the entire width of the sheets (2000 mm) perpendicular to the extrusion direction. The Δn values were measured across the width of the sheets (H) of the strips central part every 20 mm to avoid the influence of edge effects associated with cutting.

Figure 1 presents the results of these tests. The initial samples had an optical anisotropy associated with extrusion and calendar effects, with the value Δn_0. Meanwhile, these data on the nonuniformity of the BR of different monolithic PC sheets do not indicate the "poor" quality of the studied materials because the values of this nonuniformity are small compared to the maximum possible value for PC, which is 0.106 [3].

[3] GOST 3519-69. USSR State Standard. Optical glass. Method for determination of stress birefringence on polarimeter. Moscow: Izd. Standartov, 1969 (in Russ.).

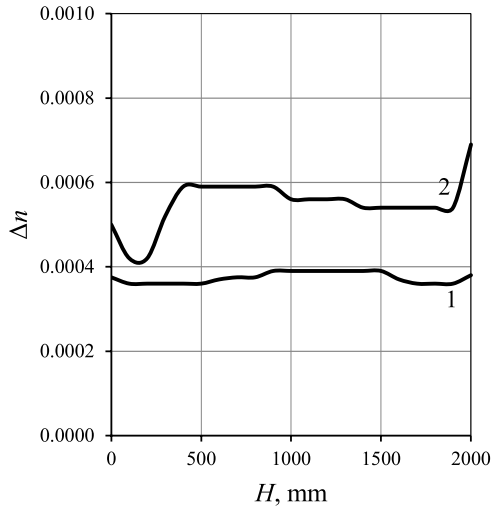

Fig. 1. Change in the value of Δ*n* along the width of monolithic polycarbonate sheets for two industrial samples without specifying their manufacturer.

The recorded changes in BR are not related directly to the nonuniform thickness of the samples because the value of thickness (δ) was considered when calculating the value of Δ*n* (Eq. 6). On the other hand, the thickness nonuniformity itself can cause thermal and deformation stress indirectly. Explicit edge effects are due to the special conditions of forming the sheet edges and their mechanical processing, including cutting.

Next, this study examined the stress state of PC samples of monolithic sheets subjected to external uniaxial loading using optical-polarization methods. At the initial stage, the dependence $\sigma = f(\varepsilon)$ of the PC samples was studied over a wide range of stress and strain. Figure 2 presents a diagram of uniaxial loading (curve 1), which describes the dependence $\sigma = f(\varepsilon)$ of the PC samples. The nature of stress growth is typical for loading diagrams of rigid amorphous PC: elastic deformation at σ up to 20–30 MPa is accompanied by noticeable plastic deformation at $\sigma > 30$ MPa, which ends with specimen destruction at stress $\sigma_d = 61.5$ MPa.

When analyzing the stressed state of the loaded samples, the relaxational decrease in the stress attained upon deformation should be considered when the sample is kept for some time at a constant deformation. This decrease is characteristic of polymers. In this regard, equilibrium stress was used in further calculations (curve 2 in Fig. 2), which was established after keeping the loaded samples at constant deformation (creep) for 600 s (Fig. 3). At stresses below 30 MPa, which is of interest in this study, this relaxation decreased when σ is small: curves 1 and 2 in Fig. 2 at this stress are the same. At stresses exceeding 30 MPa, the relaxation character of the dependences $\sigma = f(\tau)$ becomes more pronounced (Fig. 3). At stresses above 50 MPa, the samples were destroyed in less than 600 s (in Fig. 2, these stress values corresponded to the dashed section on curve 2).

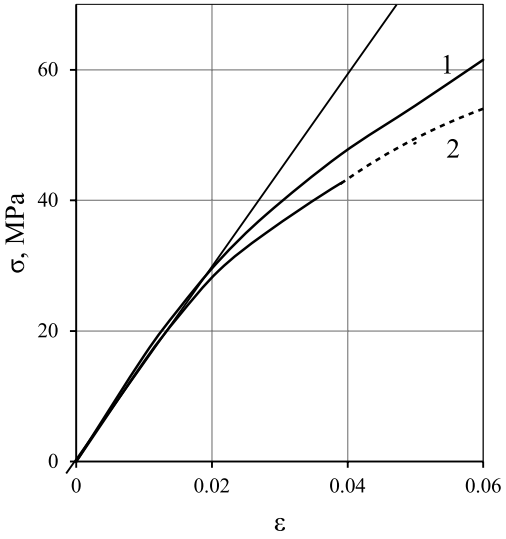

Fig. 2. Stress–strain diagrams for polycarbonate (strain rate 50 mm/min): (1) measurement during deformation and (2) measurement after relaxation for 600 s.

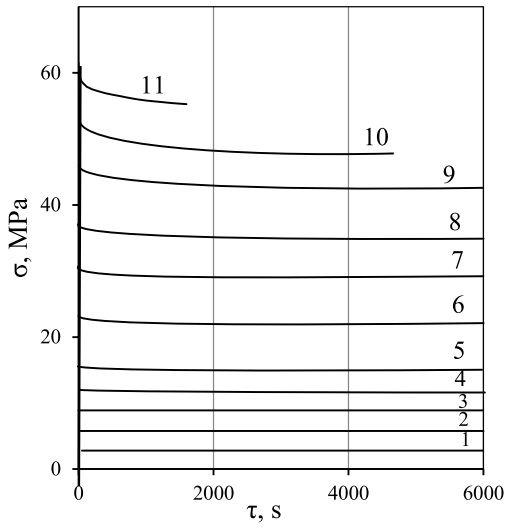

Fig. 3. Stress (σ)–relaxation time (τ) dependences at constant deformation of samples ε: (1) 0.0015, (2) 0.003, (3) 0.005, (4) 0.007, (5) 0.010, (6) 0.015, (7) 0.020, (8) 0.028, (9) 0.039, (10) 0.050, and (11) 0.063.

During further optical-polarization tests of the samples, the stress did not exceed 30 MPa. During these experiments, the strain rate was reduced to 1 mm/min to eliminate the uncertainty caused by

relaxation processes. The stress values recorded in this case (curve 2 in Fig. 2) coincided with curve 1. The strain ε did not exceed 0.5%, which minimized the effects of creep. Under these conditions, the loading rate had little effect on the stress.

The BR method is the most developed and frequently used optical technique for studying the photoelasticity of polymeric materials. Figure 4 shows the results of measuring the standard Δn value of the samples (GOST 3519-69) under the action of various specified external stress. The original sample (with residual BR $\Delta n_{res} = 0.00039$) was loaded stepwise to achieve a specified external stress during these tests. The loading was stopped at a given σ_i, and Δn_i was measured. The loading continued up to σ_{i+1}, and the value of Δn_{i+1} was determined up to 30 MPa.

The analysis of photoelasticity dependence suggests a linear relationship between the Δn value of the sample and the stress applied to it in the studied stress range. The dashed straight line in Fig. 4 describes the relationship between the external stress and Δn at $\Delta n_{res} = 0$.

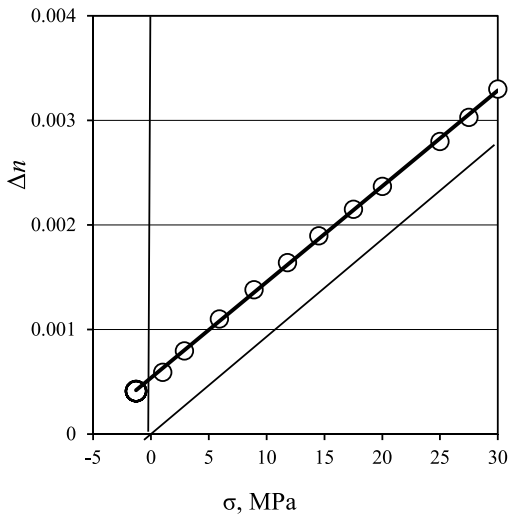

Fig. 4. Diagram describing the relationship between the birefringence value in the loaded polycarbonate specimens (conditions as in Fig. 2) and the stress applied to them (σ, MPa). The dashed line indicates the Δn values reduced to $\Delta n_{res} = 0$.

$$\sigma' = a_1 \Delta n \quad (7)$$

where σ' is the stress in the sample at $\Delta n_{res} = 0$ in MPa, and a_1 is a constant (a_1 = 11000 MPa). Equation (7) can be used as a calibration formula when estimating the stress state in the studied monolithic PC sheets. Considering the additional residual stress of the original sample (σ_{res}), which is added to the applied external stress,

$$\sigma - \sigma_{res} = a_1(\Delta n - \Delta n_{res}) \quad (8)$$

where σ is the stress applied to the sample; σ_{res} and Δn_{res} are the residual stress (MPa) and initial BR of the original unloaded sample ($\Delta n_{res} = 0.00039$).

In accordance with Eq. 8 in Fig. 4, the point corresponding to the BR of an unloaded sample is situated on the trend line describing the linear function $\Delta n = f(\sigma)$ and extrapolated to the region of negative stress. This is because the residual stress in the sample is compressive compared to the pulling stress applied to the sample. Equation 8 shows that σ_{res} for the studied sample at the point of the BR measurement is approximately 0.9 MPa (the deformation of the sample was less than 0.05%).

This technique for assessing the stress state makes it possible to characterize the product quality quantitatively. However, this method has the following disadvantages:

- the discreteness of BR measurements and the complexity of assessing the inhomogeneity on very small and large areas of the product (diameter of a light beam passing through the sample is from 1 to 3 mm);
- tests on large areas are laborious and lengthy, and it is difficult to assess the changes in product tension during its loading by this method;
- the need to cut samples from the product;
- the sensitivity of this method in the region of low stress is low.

The phenomenon of photoelasticity caused by BR in polymeric products under stress can be used to measure Δn. The nondestructive method of samples IICP analysis is based on the same physical principles. However, the area of the sample analyzed by this method is limited only by the size of the polarizer. As mentioned above, the source of polarized light was a computer monitor tuned to white light. This allows continuous real-time recording of the changes in the stress state over large areas, including changes in the process of external mechanical or thermal action on the product. The product does not need to be cut into samples for such tests.

Figure 5 shows the IICP of an unloaded sample of a monolithic PC sheet. The presence of color on the IICP of the original sample at σ = 0 was attributed

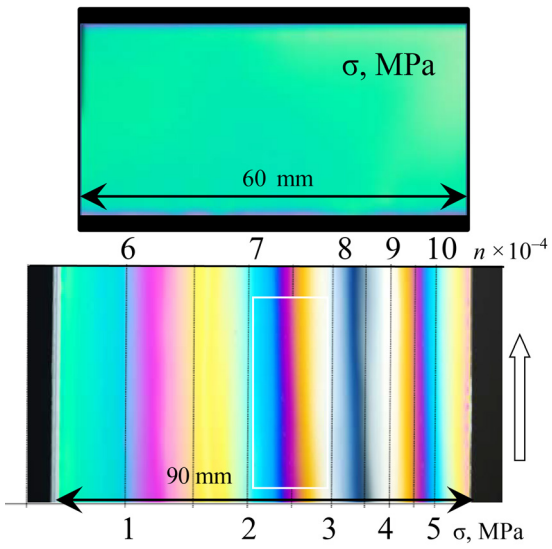

Fig. 5. Interference image in crossed polaroids of the initial sample (σ = 0 MPa) and the interference spectrum of its loading (the arrow indicates the direction of loading).

to the presence of BR, and consequently, the residual stress σ_{res}. Note that this color depends on the value of σ_{res} and the PC sheet thickness. In the full absence of BR in the sample, polarized light should not pass through the analyzer located perpendicularly to the polarizer. Therefore, the samples and background around the IICP of the original sample in Fig. 5 should be black.

The inhomogeneity of the original sample color at σ = 0 MPa (the appearance of a yellow tint in the right part of the IICP of the original unloaded sample) indicates the inhomogeneity of its Δn_{res} and σ_{res}. In addition to this "centimeter" inhomogeneity in the IICP color, the "millimeter" inhomogeneity, i.e., the change in color (from green to blue and then to pink) at the lateral edges of the sample. This reveals the residual stress that has arisen when cutting the sheet into strips, which cannot be fixed when measuring the BR. This should be considered when studying the IICP.

The step loading of the samples was carried out with the simultaneous fixing of stress values (σ_i) and interference images of the sample (similar to the step loading described above when determining Δn_i). As a basis for evaluating the change in photoelasticity in the course of loading, it is customary to measure the number of isochromatic bands (N) that appear on the sample IICP. Stress σ in the working part of the sample is calculated using the following formula [13]:

$$\frac{\sigma}{\sigma_0} = \frac{N}{\delta} \qquad (9)$$

where δ is the sheet thickness; σ_0 is the division value of the strip on the IICP, which was determined in the calibration experiments by comparing the calculated stress with the observed optical effect. Mathematical programs have been developed to interpret the IICP samples [13].

This study used the superimposing identical isochromic lines of many photographs of a loaded sample IICP taken at different applied stresses in the region of most interest from 0.3 to 5 MPa. All the points on the IICP isochromes had the same Δn value. This allowed it to combine all the IICPs obtained at different stresses affecting the studied sample into its single isochrome spectrum. Figure 5 shows a generalized picture of the colored bands (IICP spectrum). For example, the superimposed part of the sample IICP at a stress of 4.5 MPa is marked with a white contour in the IICP spectrum. However, this process can be automated. A similar IICP spectrum at different stress values can be obtained in a sample of sufficient thickness under transverse bending.

Under a load, pronounced colored isochromic bands appear at external stress above 1 MPa (not pronounced in the original samples). Figure 5 shows the actual dimensions of the sample and the resulting IICP spectrum. The stripes are oriented along the direction of the loaded sample tension. As stress increases, the isochromes "move" perpendicular to this direction, and new isochrome lines appear. Figure 5 shows the applied external stress σ_i (in MPa) and the corresponding values of Δn_i.

The distances (periods) between the isochromic lines of different orders Δi (where i indicates stresses of 1, 2, 3, 4, and 5 MPa) on the IICP spectrum (Fig. 5) become narrow with increasing acting stress according to Eq. 9. Figure 6 shows the dependence of these distances on the stress placed on the sample. The dependence is regular and adequately described by the hyperbolic function $\sigma_i = f(1/\Delta i)$.

This made it possible to use dependencies similar to Eq. 8 for BR and extrapolate their values to negative residual stress. In this case, the function $\sigma = f(1/\Delta)$ can be described as follows:

$$\sigma - \sigma_{res} = a_2(1/\Delta - 1/\Delta_{res}) \qquad (10)$$

where σ is the stress applied to the sample; σ_{res} and Δ_{res} are the residual stress (MPa) and Δ is the residual period in an original unloaded sample; k_2 is a coefficient depending on the image scale (in the present case, a_2 = 0.014 MPa). In the case under consideration, $\sigma_{res} \neq 0$ and $1/\Delta_{res} \neq 0$ in Fig. 6.

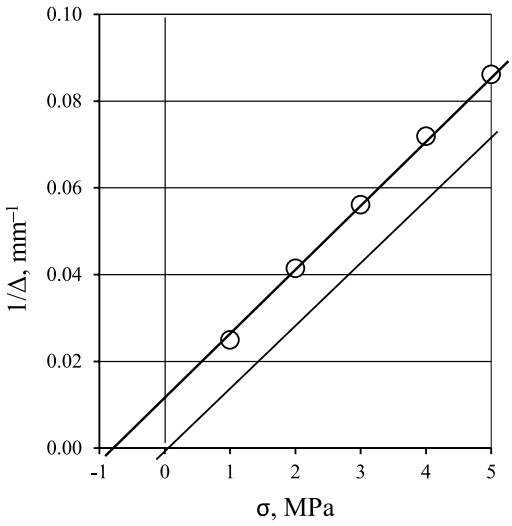

Fig. 6. Relationship between the Δ values in mm on spectrum of interference images of samples in crossed polaroids of the loaded polycarbonate samples and the stress applied to them (σ, MPa). The dashed line indicates the hypothetical $1/\Delta$ values reduced to $1/\Delta_{res}$.

This dependence can be universalized using relative units, e.g., Δ_1/Δ:

$$\sigma - \sigma_{res} = a_3(\Delta_1/\Delta - \Delta_1/\Delta_{res}) \quad (11)$$

where Δ_1 is the calculated value Δ = 78 mm from 0 to σ_1 = 1 MPa in Fig. 6; $a_3 = a_2\Delta_1 \approx 1$ (a_3 = 1.1 MPa). In this case, $\Delta_1/\Delta = 1$ at σ_1 = 1 MPa in Eq. 1. However, the analysis result will not change in this case.

The linearity of this dependence makes it possible to estimate the magnitude of residual stress in the sample, as shown in the analysis of the dependence, $\Delta n = f(\sigma)$, by extrapolation to the region of negative values of σ (compressive stress). In this case, the value of σ_{res} was 0.7 MPa, which is close to the value of σ_{res} obtained from the analysis of the stress state using the BR value of Δn_{res} (0.9 MPa). The average stress, 0.8 ± 0.1 MPa, is small, corresponding to <0.05% elastic deformation of the PC sheet. This does not exceed the usual thermal deformation of PC sheets upon their operation, which can be considered safe. Previous studies [1, 2] showed that less than 10 MPa is the safe stress that ensures the long-term operation of PC sheets and products made from them.

The resulting IICP spectrum can be used to determine the stress in the studied PC samples based on their interference images. To accomplish this, it is sufficient to combine the same isochromic lines of the sample IICP and IICP spectrum and determine the coordinate of the test sample center from the IICP spectrum length. The latter is equal to the sum,

$$x_k = \sum \Delta_i = \sum_{n=1}^{k} \left(\frac{1}{n^a}\right) \quad (12)$$

where x is the test sample coordinate on the IICP spectrum; n = 1, 2, 3 …, which corresponds to σ in MPa in Fig. 6; a is the exponent approximately equal to 1 (a = 1.1). The difference between exponent a and 1 is due to the residual stress in the sample. Figure 7 presents a diagram that graphically describes Eq. 12. The diagram can be used to determine the stress when analyzing the IICP spectrum shown in Fig. 5. Equation 12 makes it possible to extrapolate this diagram to higher stress in the elastic region. In Fig. 7, the dependence is extrapolated to 10 MPa. Previous studies [1, 2] reported that stresses less than this value are acceptable during the warranty period of PC sheets.

However, the picture of the IICP isochromes depends on the stress and other factors, e.g., on the sheet thickness and its thickness variation, the slope of the product sections. This may reduce the reliability of the quantitative assessment of its stress state. On the other hand, this problem for initial sheets or specific identical products made of them can be solved by developing a database of reference IICP

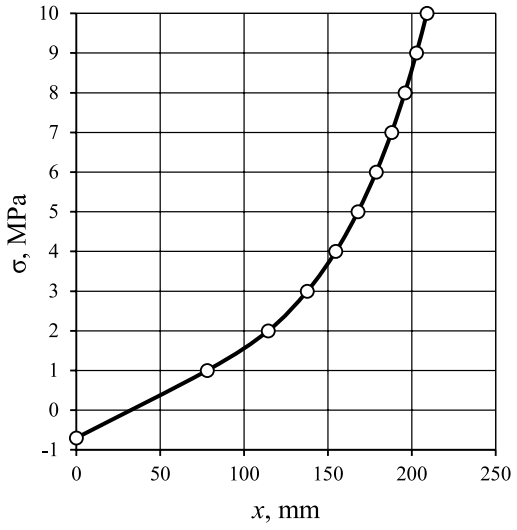

Fig. 7. Graphic depiction of Eq. (12). The dots are the experimental values.

spectra of quality products, as done for the automated assessment of the stress state of molded PC blanks of compact discs [15].

CONCLUSIONS

These studies showed that optical-polarization methods effectively assess the stress state of PC monolithic sheets made by various manufacturers in the Russian Federation. A quantitative relationship was established between the stress affecting PC samples, their BR values, and the characteristics of the combined spectrum of interference images of loaded samples obtained in polarized light in crossed polaroids. Overall, the residual stress values in monolithic PC sheets can be quantified by analyzing the spectra of their interference images. The measured residual stresses do not exceed 1 MPa.

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Authors' contribution

A.V. Markov – design of the research concept, development of the experiment, discussion and analysis of the results, writing the text of the article;

V.N. Lobanov – studying the properties of samples, processing and analysis of the data obtained, discussion of the results.

The authors declare no conflicts of interest.

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