STRUCTURE AND MECHANICAL PROPERTIES

OF A DISPERSEDLY FILLED TRANSPARENT

POLYCARBONATE

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The structure and mechanical properties of a composition on the basic of polycarbonate filled with three types of dispersed particles (submicron corundum, micron boron nitride, and glass fiber ones) were investigated. It is shown that the method of sample preparation proposed ensures a uniform distribution of filler particles in them. Slight changes in the supramolecular structure of polycarbonate were seen only in the case of introduction of submicron corundum particles. No such changes occurred in the case of micron boron nitride or glass fiber particles in amounts providing an optical transparency no less than 70%, which can be explained by insufficient interaction forces between filler particles and polycarbonate molecules. It is shown that filling polycarbonate with various fillers allowing the retention of its optical transparency does not make it possible to increase its wear resistance and softening temperature. The weight parts of dispersed filler at which the transparency does not decrease below 70 % are ~0.06, ~0.03, and ~1.0% for corundum, boron nitride, and glass fiber particles, respectively.

1. Introduction

Currently, in creating glazing structures for various purpose, e.g., for cabins of vehicles and show-windows, polymer glasses, such as polycarbonate and polymethil methacrylate (plexiglass) are increasingly being used. This is explained by the higher durability of such glasses, in comparison with the inorganic glass, at a lower weight and the same high optical

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transparency [1-3]. However, the application fields of polymer glasses are limited by insufficiently high values of some their other characteristics, e.g., the scratch resistance, softening temperature (~140-150°C for polycarbonate), and tensile strength [4-6]. Thus, the low scratch resistance (wear resistance) of polycarbonate glass, which is used for glazing vehicles, results in its decreased transparency during operation by more than 30% and makes its further operation impossible [4, 7, 8]. In general, two basic approaches are used to improveme the characteristics of the polymer glasses mentioned, without changing the chemical composition of the link of polymer molecules:

- application of coverings [5, 8] with high adhesion to the polymer glass, high wear resistance, high resistance to the action of ultraviolet and cyclic temperature differences and with preservation of its optical transparency not lower than 85-95%;

- application of disperse fillers [2, 6, 9, 10].

The second approach, contrary to the first one, allows one to improve not only the wear resistance and thermal stability of the surface of polymer glass, but also some mechanical characteristics, e. g., the hardness and maximum breaking tensile stress, especially on using nanosize filler particles [7-11]. In [11], it is shown experimentally that the introduction such particles increases the elastic modulus and dissipative characteristics of polymer more than the addition of microparticles with the same volume fraction. The main restriction of such an approach is the reduction in its transparency [12, 13]. For glazing vehicles, the transparency should not be lower than 70% [14]. To ensure the transparency required and to simultaneously increase the wear resistance, thermal stability, hardness, and durability of polymer glass, it is expedient to use submicron (of characteristic size no more than wavelengths in the optical range) filler particles or particles with a refractive index close to that of polymer glass [13]. We should note that a high light scattering can be caused not only by the characteristics (the size and refractive index) of filler particles, but also by optical inhomogenities, e.g., the cavities between particles and the polymeric matrix [12, 15] caused by poor adhesion between the particles and polymer [16]. To increase this adhesion, the filler and polymeric matrix are modified, for example, by creating an intermediate polymer layer on particle surface [17] or by activating their surface [10, 16]. All these measures increase the weigh fraction of dispersed filler particles in the polymeric matrix only slightly, and, accordingly, only somewhat increase the mechanical characteristics of such a glass, but they do not remove the main restriction of the approach considered. Therefore, an important characteristic for practice in using such an approach to improve the mechanical characteristics of polymer glasses is the weight fraction ω_{opt} of filler particles in them that simultaneously increases their mechanical characteristics and ensures an optical transparency not lower than 70%.

The purpose of the given work is to determine, by the example of polycarbonate, the weight fraction ω_{opt} of filler particles in a polymer glass providing the highest values of some its mechanical characteristics (scratch resistance, softening temperature, maximum stress, hardness, and tensile strength) with preservation of transparency of the polymer glass not lower than 70%.

1. Sample Preparation Technique

The value of ω_{opt} in the polymer glass was determined experimentally on samples of filled polycarbonate (PC). Three types of filler particles were used:

- submicron-size (~115 nm) corundum (Al_2O_3) particles (a mix of δ and θ phases). This size is less than wavelengths of the optical range (400-760 nm), which allows one to lower the light scattering and to raise the volume concentration of these particles in polycarbonate [12, 13];

- micron-size (~1 μm) boron nitride (BN) particles;

- glass fiber particles with a characteristic size of ~100-1000 μm, whose refractive index (1.52) is close to that of polycarbonate (1.59). However, as preliminary theoretical estimations have shown [13] that, even in this case, it is possible to ensure the transparency of material not below 70% only at weight concentrations of glass fibers in polycarbonate not exceeding ~1%.

To determine the value of ω_{opt} at which the mentioned mechanical characteristics of dispersedly filled polymer glass reach the highest values, the weight fraction ω of filler particles in PC samples with corundum (samples 1) was

varied within the limits of 0.05-0.5%, those with boron nitride (samples 2) — 0.01-3%, and those with glass fibers (samples 3) — 0.05-1%.

The ratio of weight fractions was ensured using the weight method with an accuracy of 0.005 g.

Before introduction of all the types of particles into polycarbonate, they were preliminary prepared. Agglomerates of Al_2O_3 nanoparticles were dispersed in a suspension with isopropyl alcohol in an ultrasonic bath (of power 40 W) no less than 30 min. The sedimentation of dispersed particles on PC granules of mark PC-007 (THAT 2226-173-00203335-2007) was carried out directly in the ultrasonic bath with isopropyl alcohol. The weight fraction of Al_2O_3 nanoparticles settled on PC granules was determined by weighing with an accuracy of 0.005 g, and then the granules were dried at a temperature $120 \pm 5°C$ to remove the alcohol from them.

The agglomerates of BN and glass fiber particles were dispersed in a unipolar corona discharge of atmospheric pressure, which also led to the formation of hydroxyl groups on the surface of these particles [18]. This increased the adhesion of the particles to polycarbonate [16, 18] and, as a consequence, increased the transparency of samples with preservation of the volume concentration of particles. The parameters of the gas discharge were as follows: pressure — atmospheric; voltage 10 ± 0.5 kV; interelectrode gap 5 ± 1 mm; barrier— a heat-resistant glass; humidity of air — no less than 60%; dispersion time 15 ± 2 min.

The distribution uniformity of nanofiller particles in the polycarbonate matrix was achieved by preparing a 15% solution of PC together with filler particles in methylene chloride, with additional subsequent dispersion of filler particles in a PC solution in an ultrasonic bath up to the full evaporation of solvent. We should note what, to ensure the distribution uniformity of submicron and micron filler particles by using traditional methods of their introduction into polycarbonate, for example, mixing in an extruder [3], is difficult owing to the high viscosity of PC melt. Besides, the filler particles tend to aggregate [3, 16] in the polymer melt, which results in lower values of the mechanical characteristics of polymer composition than expected. Disk-shaped samples of filled polycarbonate (of thickness 2 mm and diameter 52 mm) were made by hot pressing (temperature $280 \pm 10°C$ and pressure 7 ± 0.5 atm) after the evaporation of solvent.

Before measuring the mechanical and optical characteristics of the samples, they were normalized by removing the internal residual mechanical stresses arising during their preparation [19]. To do this, each sample was first heated to $120 \pm 5°C$ in a muffle furnace from the reference temperature of $23 \pm 3°C$ at a initial humidity of 55 ± 5%; then, the sample was held at a temperature $120 \pm 5°C$ during 300 ± 20 min and slowly cooled to an ambient temperature of $23 \pm 3°C$ during 24 h. The cooling was also carried out in the muffle furnace.

2. Experimental Results and Their Discussion

2.1. Investigation of material structure

The material structure was examined on a Carl Zeiss AURIGA Cross Beam electron microscope with EDD Inca X-Max 80 mm². The characteristic microphotos of cleavage fragments of samples are shown on Fig. 1. The cleavage were obtained after cooling the samples of filled PC in liquid nitrogen.

Changes in the supramolecular structures of filled polycarbonate samples were observed only for samples 1 (see Fig. 1) at $\omega \approx 0.15$%; hence, greater changes in the mechanical characteristics could be expected exactly in these samples [20]. The absence of structurization of the polymeric matrix in samples 2 and 3 at all values ω in the indicated ranges was caused by insufficient interaction forces between filler particles and polycarbonate molecules [21], in contrast to those in samples 1, in which the high interaction forces were caused by dimensional effects [22, 23]. We should note that more expressed changes in the supramolecular structure of polycarbonate filled with nanoparticles have been observed in films obtained from a solution without any thermal processing [20, 24]. Most likely, in manufacturing samples with the use of their thermal processing, the bonds between filler particles and PC matrix are broken, which indirectly points to their physical nature [25]. A preliminary dispersion and activation of particles raised their wettability with polycarbonate (see
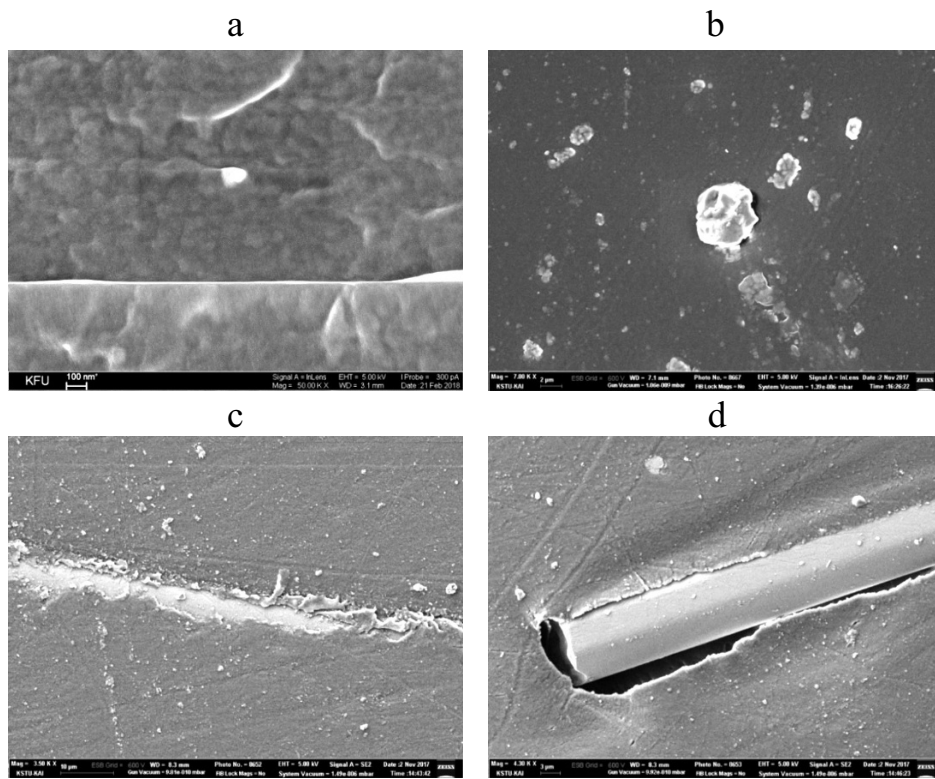
Fig. 1. Microphotos of the surface of PC samples filled with submicronic Al_2O_3 particles (a), micron BN particles (b), and glass fibers activated (c), and nonactivated (d) in plasma discharge.
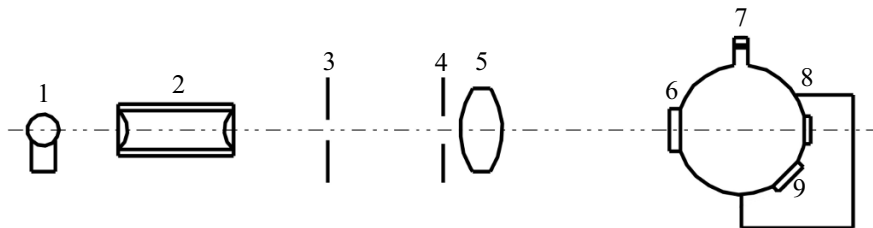

Fig. 2. Diagram of measuring the light transmission factor and optical transparency of samples filled with polycarbonate: 1 — light source, 2 — collimator, 3 — entrance diaphragm, 4 — aperture diaphragm, 5 — lens, 6 — sample, 7 — photometer, 8 — light trap, 9 — reflecting standard sample.

Fig. 1c), and, as could be expected, increased transparency at a constant concentration of filler particles. This is especially important for particles whose sizes are comparable with or exceed the wavelength. The voids formed on the interface of the polymeric matrix and filler particles with a poor wettability (see Fig. 1d) increase the light scattering [12], which decreases the transparency of polymer glass [16].

2.2. Investigation of optical characteristics

The diagram of measuring the optical transparency and light transmission factor of the filled polycarbonate samples is shown on Fig. 2. In the measurements, an integrating photometric sphere of diameter 200 mm was used.
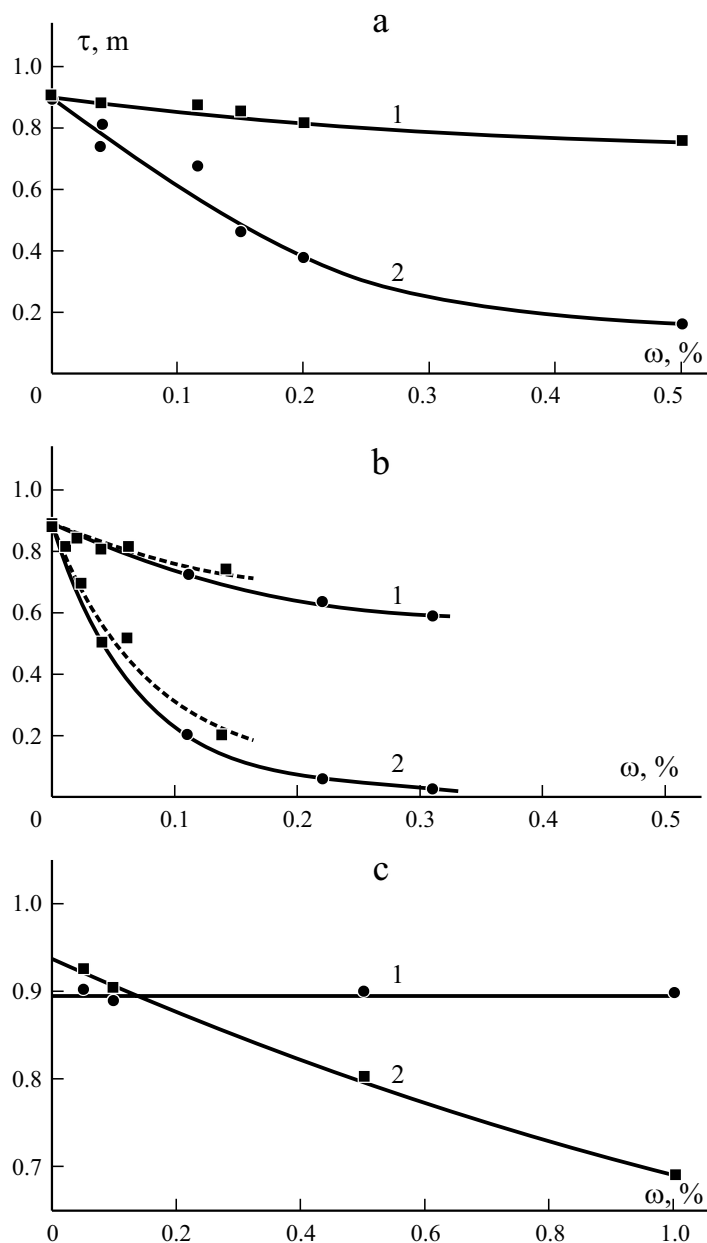
Fig. 3. Typical relations for the transmission factor τ (1) and optical transparency M (2) of PC samples filled with Al_2O_3 (a) and BN (b) particles and glass fibers (c) as functions of ω. Points — experiment and lines — approximation. Other explanations in the text.

The light transmission factor and optical transparency were determined on all the surface of samples of the filled polycarbonate with an error of ~10%. Typical relations for the light transmission factor τ and optical transparency M as function of filler concentration ω in samples are shown on Fig. 3. On Fig. 3b, the continuous curves correspond to the PC samples filled with nonactivated BN particles, and the dashed ones — to those with particles activated in the corona discharge. The τ and M curves on Fig. 3c are given for PC samples filled with glass fibers activated in the corona discharge.

The weight fraction ω of submicronic corundum particles at which the transparency of composite glasses was ~70% was ~0.06% (see Fig. 3). At such a weigh fraction of the particles, the average distance between them in the polymeric

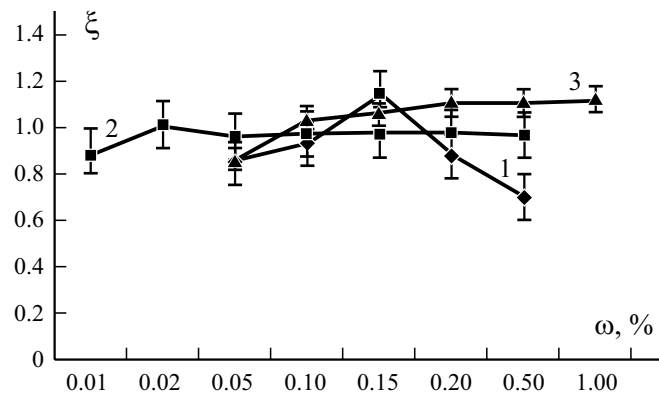Fig. 4. The relative maximum lengthening ξ of samples 1 (1), 2 (2), and; 3 (3) vs. the weight fraction ω of filler particles.

matrix at their homogeneous distribution was ~10 μm, which was 100 times more than that between 115-nm corundum particles.

The weight fraction of BN particles up to which the transparency of composite glasses was not below 70% was ~0.03% (see fig. 3). Thus, the average distance between the filler particles in the polymeric matrix at their homogeneous distribution was ~100 μm, which 20 times exceeded that of 5-μm BN particles. The activation BN of particles in the corona discharge before their introduction into the material increased its optical transparency insignificantly. The basic contribution to the reduction in transparency brings in the light scattering on particles of micron size, but the contribution of the optical inhomogeneities arising on particle-polymer interfaces is less significant.

The light transmission factor τ practically did not depend on the glass fiber concentration in polymer samples owing the close refractive indices. However, the transparency of such a composition, as expected, decreased sharply with growing weight fraction (more than 1%) of glass fibers in the polycarbonate matrix (see Fig. 3), which was caused by the increased light scattering on the optical inhomogeneities arising on particle-polymer interfaces [13].

Thus, there exists a limiting concentration ω_{max} of filler particles, both of submicron and micron sizes, in polymer glasses on exceeding which the optical transparency drops below the allowable one: $\omega_{max} \sim 0.06\%$ for submicron particles, $\omega_{max} \sim 0.03\%$, for micron particles, and $\omega_{max} \sim 1\%$ for micron particles with a refractive index close to that of polymer glass.

2.3. Investigation of mechanical characteristics

The Martens hardness of filled composites was determined according to ISO14577-1 by using a DUH-211 Shimadzu ultramicrotester with a Vickers indenter. Measurements were carried out at different indenting forces and indenting time of 30 s. Within the limits of ~10% measurement error, the hardness of filled samples did not depend on ω. The averaged hardnesses of samples 1, 2, and 3 were 61.35, 61.31, and 61.35 mN, respectively, but the hardness of unfilled PC was 61.3 mN, i.e., actually the same.

The tensile strength of samples with different values of ω was carried out on a Shimadzu A650 kNX universal desktop test machine with the use of a Shimadzu Trapiziumx software. Results of the tests are shown on Fig. 4.

The maximum stress σ_{max} of PC samples filled with BN particles did not depend on the weight fraction ω of the particles within the limits of 10% measurement error. The values of σ_{max} averaged over samples with different ω were 39.90 ± 2.9 N/mm^2 for the unfilled PC, 42.83 ± 3.2 N/mm^2 for samples 2, $\sigma_{max} = 63.34 \pm 4.75$ N/mm^2 for samples 1 at $\omega_{opt} = 0.15 \pm 0.02\%$, and 60.21 ± 1.75 N/mm^2 for samples 3 at $\omega \geq 0.2 \pm 0.02\%$. It is likely that a higher content of corundum particles (samples 1) in polycarbonate reduces the mobility of polymer chains and, as consequence, decreases

TABLE 1. Measured Scratch Rsistance of Filled Polycarbonate Samples

Filler type	ω, %	Load at the appearance of scratchs, kgf
Unfilled PC	0	1 ± 0.1
Al_2O_3	0.05	1 ± 0.1
Al_2O_3	0.2	1 ± 0.1
Al_2O_3	0.5	2 ± 0.1
Al_2O_3	1	3 ± 0.1
BN	0.01	1 ± 0.1
BN	0.02	1 ± 0.1
BN	0.04	1 ± 0.1
Glass fibers	0.5	1 ± 0.1
Glass fibers	1	1.5 ± 0.1

the strain at break [25]. For samples 2, as expected, the mechanical properties poorly depended on the weight fraction of particles, whose presence decreased the optical transparency. The increased maximum stress in sample 3 at $\omega \geq 0.2 \pm 0.02\%$ could be caused by influence of the tensile strength of glass fibers themselves [26].

The wear resistance of samples of the filled PC was investigated on a "Konstanta-TS2" device with a U-shaped wire indenter of diameter 1.6 mm with a 3.25-mm curvature.

The error of the filler weight fraction in PC samples presented in Table 1 is within the limits of 12%. At $\omega \geq 1\%$, the scratch resistence of samples 1, contrary to that of samples 2 and 3, increased considerably — three-fold against samples 2 and 3. Most likely this, was caused by higher interaction forces between the submicron particles and polymer molecules than in the case of micron ones [22, 23, 26, 27].

2.4. Investigation of thermal characteristics

Two methods were used for this purpose.

Method 1 — determining the softening temperature by the Wick method. The softening temperature was measured on a DMA 242 E Artemis (Netzsch) dynamic mechanical analyzer. The range of working temperatures of the equipment is from −170 to 600°C, the heating rate is from 0.01 to 20 K/min, the accuracy of prescribed temperature is 0.1°C, and the accuracy of strain measurements was 0.1 μm. The parameters at which the softening temperature was measured were as follows: indenter diameter 1 ± 0.2 mm, indenter area 0.79 ± 0.1 mm^2, indentation force 7.85 ± 0.5 N, calculated load 10 ± 0.1 MPa, and indenter penetration depth 1 ± 0.1 mm.

The softening temperatures of all dispersedly filled PC samples, relative to that (~156°C) of the unfilled reference, PC sample, were within the 10% measurement error at preservation of the optical transparency not lower than 70%. A growth in the softening temperature began at filler weight fractions in PC considerably exceeding the lower limit of sample transparency ~160°C at $\omega = 0.25 \pm 0.2\%$ for the PC filled with corundum particles, ~161°C at $\omega = 3 \pm 0.3\%$ for the PC filled with BN particles, and ~160°C at $\omega = 1 \pm 0.1\%$ for the PC filled with glass fibers. These results can be explained by the interaction between filler particles, which grew with their concentration in the material.

Method 2 — measuring the glass-transmission temperature by the method of dynamic mechanical analysis. This temperature was measured on a DMA Q800 (TA Instruments, USA) dynamic mechanical analyzer. The sample was tested in bending in the dynamic regime at a constant heating rate of 5°C/min. The deformation amplitude was 15 μm and loading frequency 10 Hz. The range of working temperatures of the equipment is from −170 to 600°C, the heating rate from 0.01
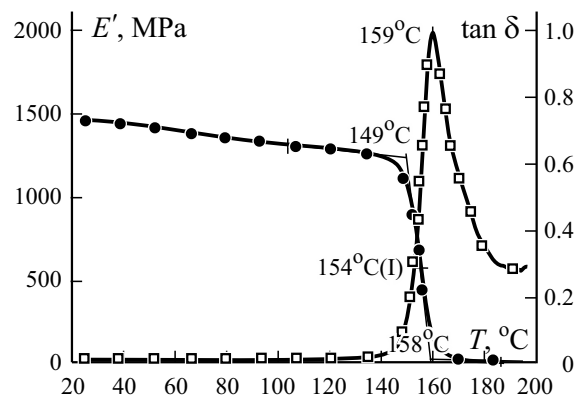
Fig. 5. Thermomechanical curve of a thermoplastic PC sample filled with corundum particles with ω = 0.06%. E' is the storage modulus.

to 20°C/min, the accuracy of prescribed temperature 0.1°C, and the accuracy of deformation measurements 0.1 μm. An example of the thermomechanical curve is shown on Fig. 5.

The glass-transmission temperatures all the dispersedly filled PC samples coincided (within the limits of their 10% measurement error) with that (~157°C) of unfilled PC at all the fractions of filler particles considered.

Conclusion

Results of this research have shown that it is possible to raise some mechanical characteristics (scratch resistance, maximum stress, and tensile strength) of dispersedly filled PC with retention of allowable optical transparency (not less than 70%) by introducing filler nanoparticles (corundum ones of an average size less than the wavelength) or microparticles (for example, glass fiber ones) whose refractive index is close to that of PC.

The highest value of the ratio between the maximum stress of PC samples filled with corundum particles and that of unfilled PC is 1.58 at ω_{opt} = 0.15 ± 0.02%. A higher concentration of nanoparticles in PC reduces the mobility of polymer chains and, as consequence, decreases the strain at break. We should note that, on introduction of corundum particles into PC, structurization of the supermolecular structure of polymer composition occurs, which is caused by rather high interaction forces between the filler particles and PC molecules in contrast to those in samples with other filler particles. The difference between them and the corresponding maximum stresses in the case of glass fiber is ~1.5 at $\omega_{opt} \geq 0.2 \pm 0.02\%$. The considerable increase in such characteristics as the scratch resistance and softening temperature of dispersedly filled PC corresponds to the weight fractions of different fillers at which the optical transparency is below the allowable 70%. Thus, a three-fold increase in the scratch stability compared with that of unfilled PC was achieved using corundum nanoparticles with $\omega \geq 1\%$.

To light scattering in filled PC samples can be lowered by formation of a transition layer, a polymer shell, on the surface of submicron filler particles, for example, polystyrene (with a refractive index refraction 1.58), by selecting its parameters — the thickness and refractive index [12, 15].

The approach to obtaining samples of dispersedly filled polycarbonate from a solution proposed in this work allows one to achieve a homogeneous distribution of filler particles in the polymeric matrix, which is confirmed by the results of scanning electronic microscopy. It lowered the agglomeration of dispersed nanoparticles in polycarbonate and led to more reliable results for the influence of weight fraction of such particles on its mechanical characteristics.

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