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ANALYSIS OF MICROSTRUCTURE OF LAMINATED POLYMER COMPOSITE MATERIAL OF METAL COMPOSITE OVERWRAPPED PRESSURE VESSEL

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The microstructure of a layered polymeric composite material used in the construction of a metal composite overwrapped pressure vessel is investigated. The purpose of this work was to evaluate the parameters characterizing the structure of a laminate polymer composite material. Due to their technological and structural features, laminated polymer composite materials have a number of operational disadvantages that lead to a reduction in the overall level of strength characteristics. From the different zones of the nine-layer composite shell of the metal composite overwrapped pressure vessel, four vertical flat samples of the composite material for the manufacture of thin sections were cut out. The method of electron-scanning microscopy was used. The analysis of the percentage confinement of fibers in the matrix was carried out. The structure of the layered polymeric composite material is uniform with the presence of a dispersion of distances between the fibers. The analysis of porosity in a composite material was carried out. The analysis of the structure of composite materials with different porosity has shown that with increasing pore area and their number, the strength characteristics of composite tapes and reinforcing fibers decrease. Using the "mixture rule" and "polydispersity model", the values of the effective modulus of elasticity of the composite material are estimated. It is determined that the modulus of elasticity of the composite material in the zone of the flange of the composite shell is less than at the equator. A complex evaluation of the quality of a laminate polymer composite material used in the structure of a metal composite overwrapped pressure vessel was carried out. The obtained results of inhomogeneity of the mechanical properties of the composite shell are necessary for design calculation of the stress-stain state of metal overwrapped pressure vessels.

Keywords: microstructure, carbon fibers, pores, modulus of elasticity, composite material, metal composite vessel.

АНАЛИЗ МИКРОСТРУКТУРЫ СЛОИСТОГО ПОЛИМЕРНОГО КОМПОЗИЦИОННОГО МАТЕРИАЛА МЕТАЛЛОКОМПОЗИТНОГО БАКА ВЫСОКОГО ДАВЛЕНИЯ

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Исследована микроструктура слоистого полимерного композиционного материала, применяемого в конструкции металлокомпозитного бака высокого давления. Целью исследования являлась оценка параметров, характеризующих структуру слоистого полимерного композиционного материала. В связи с технологическими и конструктивными особенностями слоистые полимерные композиционные материалы имеют ряд недостатков при эксплуатации, которые приводят к снижению общего уровня прочностных характеристик. Из разных зон 9-слойной композитной оболочки металлокомпозитного бака высокого давления было вырезано 4 вертикальных плоских образца композиционного материала для изготовления шлифов. Исследования проводились с использованием электронно-сканирующей микроскопии. В ходе исследований были получены изображения микроструктуры композиционного материала с характерным высоким уровнем контраста между волокном и матрицей. Проведен анализ процентного содержания углеродных волокон в матрице. Определено, что структура слоистого полимерного композиционного материала однородна с наличием разброса расстояний между волокнами. Сравнительный анализ случайных зон микроструктуры композиционного материала указал на то, что разница в процентном содержании углеродных волокон в матрице между образцами, вырезанными с экватора, небольшая, однако имеется существенная разница между образцом, вырезанным с экватора, и образцом, вырезанным в зоне фланца. Проведен анализ пористости в композиционном материале. Анализ структу-

ры композиционных материалов с различной пористостью показал, что с ростом площади пор и их количества характеристики прочности композитных лент и армирующих волокон уменьшаются. Рассчитаны значения эффективного модуля упругости композиционного материала с использованием «правила смеси» и «полидисперсной модели». Определено, что модуль упругости композиционного материала в зоне фланца композитной оболочки меньше, чем на экваторе. Проведена комплексная оценка качества слоистого полимерного композиционного материала, применяемого в конструкции металлокомпозитного бака высокого давления. Полученные результаты неоднородности механических свойств композитной оболочки могут быть использованы при проектных расчетах напряженно-деформированного состояния металлокомпозитных баков высокого давления.

Ключевые слова: микроструктура, углеродные волокна, поры, модуль упругости, композиционный материал, металлокомпозитный бак.

Introduction. Layered polymeric composite materials are one of the most popular and promising materials in the space and aviation industries. Composite materials have high strength, increased stiffness and low specific gravity, which increase the service life of products [1]. The main feature of composite materials is the structure of the material, which can be formed for a specific task and provide the necessary characteristics of mechanical properties. However, due to technological and structural features, composite materials have a number of shortcomings that appear during operation: the fibers break, there is a loss of stability of the fibers in the matrix, pores and voids are formed, the matrix is peeled off and microcracks are distributed [2]. All this leads to the emergence of stress concentrators and reduction in the overall level of strength characteristics.

Fibers in the composite material have higher physical and mechanical properties than components. When the matrix in the composite material is destroyed, the stresses are redistributed to adjacent fibers [3]. The higher the fiber content in the matrix and the packing density is, the higher the modulus of elasticity and strength of the material are. Thus, the volume content of the filler (fibers) plays an important role in the composition of polymer composite materials. In this regard, there is a need for detailed consideration of the volume content of carbon fibers in the epoxy matrix, which have a significant effect on the characteristics of mechanical properties.

Another important parameter that determines the strength properties of a composite material is the porosity of the material structure. The reasons for the appearance of pores in composite materials are explained differently by researchers: some believe that pores are formed from residues of air and volatile products captured during the manufacture of prepregs; others account it for the presence of moisture in the structure of the composite, while others suggest that pores are formed as a result of both manifestations [4]. The pores in the composite material primarily influence the strength properties of the material, the fiber packing density in the matrix is disturbed (large distances between the fibers occur), there is a loss of fiber stability in the matrix and the formation of cracks at the pore boundaries. All this leads to additional stresses, which directly affects the strength characteristics of the material.

Due to these features of the structure of laminate polymeric composite materials, it is necessary to examine in detail their structural parameters.

Existing domestic and foreign methods for studying the microstructure of layered polymeric composite materials do not allow cconducting full research and providing a comprehensive assessment of the quality of the material. The standard method for determining the volume content of the reinforcing filler is based on the chemical decomposition of the ASTMD 3171 polymer matrix [5]. The main problem in determining the volume content of reinforcing filler according to this method is the assumption of complete destruction of the polymer matrix in the absence of a destructive effect on the reinforcing filler [6]. Standard methods for determining the structure of composite materials for the presence of porosity are based on determining the density of the composite and its constituent components according to ASTM D 2734 [7] or GOST 15139-69 [8]. This method determines the values of the analyzed parameter averaged over the sample volume and does not allow obtaining complete information on the geometric characteristics of the pores and their distribution over the sample surface.

The optimal solution for studying the microstructure of laminated polymeric composite materials is the use of electron-scanning microscopy [9–11]. The purpose of this work was to evaluate the parameters characterizing the structure of a polymeric laminated composite material used in the construction of a metal-composite high-pressure tank (MCHPT) using electron scanning microscopy to determine: the volume content of fibers in a composite tape, the porosity, the volume content of fibers and the effective modulus of elasticity of composite material.

Samples, methods, equipment, sample preparation. The object of the study was the microstructure of the composite shell, formed during the production of a metal-composite high-pressure tank. This tank is used in electric reactive propulsion systems of space vehicles and is intended for storage of xenon (working fuel). The MCHPT consists of a titanium liner that provides tightness, and a 9-layer power composite shell providing structural strength (fig. 1).

The composite shell is made by the method of continuous multilayer winding on the liner of a composite tape impregnated with an epoxy binder ED-I. The composite material consists of a set of differently oriented composite tapes based on T1000 carbon fibers, which, during the winding process, are laid on the underlying layers at different reinforcement angles. The structural diagram of the layered polymer composite used in the MCHPT is shown in fig. 2.
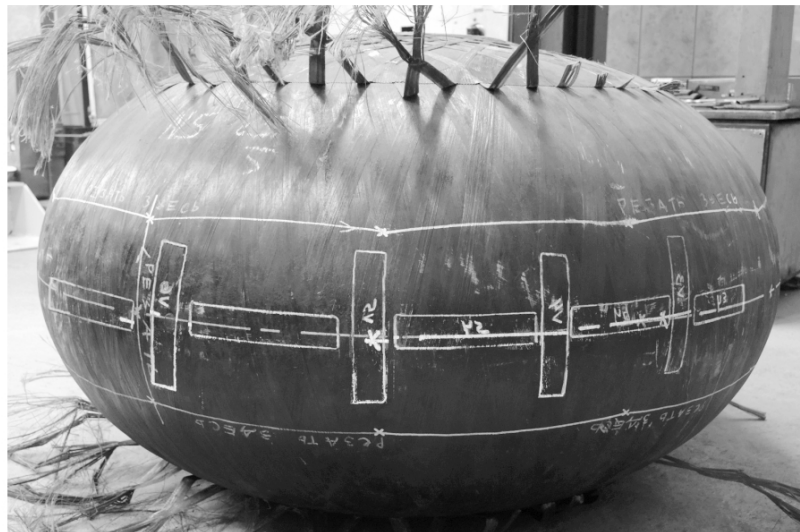
Fig. 1. Metal composite overwrapped pressure vessel with designated zones
for cutting samples

Рис. 1. Металлокомпозитный бак высокого давления с обозначенными
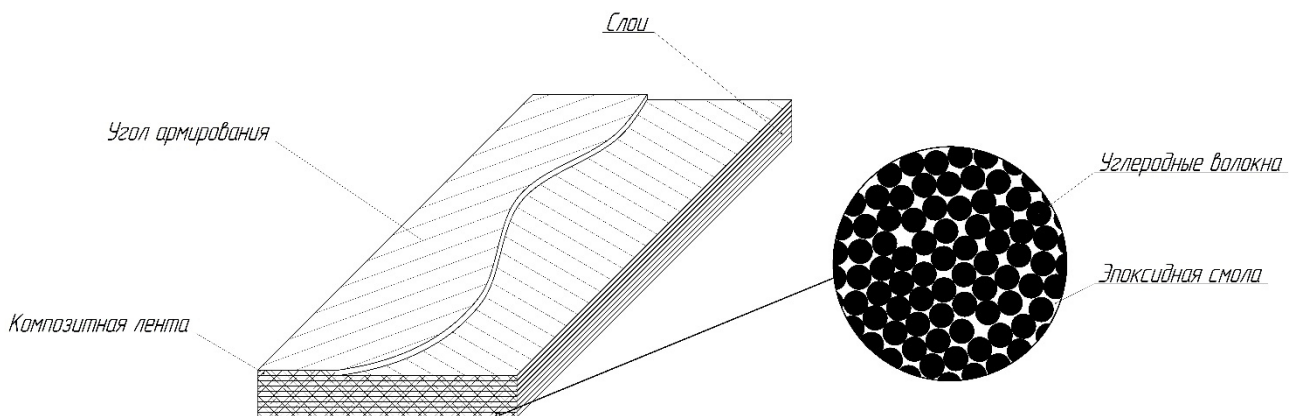зонами для вырезки образцов

Fig. 2. The scheme of unidirectional 9-layer composite material

Рис. 2. Схема однонаправленного 9-слойного композиционного материала

In the design of the MCHPT, the most suitable zone for cutting unidirectional specimens is the equatorial zone, in which the angle of laying the belt is minimal and is 10 degrees [12]. In addition, unidirectional samples were cut near the MCHPT flange. Due to the technological feature of winding of the composite tape on the liner, the shell has a variable thickness. As we approach the pole hole, the composite tape more and more overlaps the underlying layers, forming a composite shell of variable thickness, which leads to heterogeneity of mechanical properties, as well as the appearance of additional flexural forces [13]. This necessitates a detailed consideration of the microstructure of the composite material in the flange region.

Experimental studies were carried out in the laboratory of electron-structural studies of the "Center for Collective Use" of the Siberian Federal University (CCU of SFU).

From the different areas of the MCHPT 4 vertical flat samples of composite material were cut out to make polished sections. Flat unidirectional samples were cut with a linear precision saw IsoMet 5000 BUEHLER. Grinding was carried out using silicon carbide paper (grain size up to P2400), after which the polished sections were polished using diamond pastes with a gradual reduction in the grain size to 0.25 μm. The final polishing was carried out on the Beta Grinder-Polisher (BUEHLER). The samples were then cleaned in an ultrasonic bath in high purity alcohol, and dried. The samples were weighed on a Mettler ToledoXS 205 DR analytical balance (0.1 μg accuracy). The samples were pressed into a phenolic epoxy resin on a Simplimet 3000 automatic press (BUEHLER), then their cross sections were polished with diamond paste up to 0.25 μm in size.

The primary visual control of the surface of the samples was carried out with an optical microscope Nikon

LV100D. The microscope is equipped with a CCD camera (5 megapixel) for transferring images to a computer. Subsequent studies of the microstructure and morphology of the surface of the transverse sections of the samples were carried out with an electron scanning microscope (ESM) JEOL JSM 6490LV.

In the course of the studies, images of the microstructure of the composite material of the MCHPT were obtained using an ESM with a distinctive high level of contrast between the fiber and the matrix. To estimate the average value of the volume content of fibers in the matrix, measurements were made for randomly selected zones. According to the recommendations, in the study [12] we applied zoom with 30 to 100 fibers appearing in the field of view, in the case of ESM usage the optimum zoom was x2000.

Results and their discussion Determination of the volume content of fibers in a composite tape. Fig. 3 shows the microstructure of the composite material cut from the equator of the MCHPT. The image at 2000 times magnification was processed using ImageJ. (fig. 3, *b*).

From the analysis of the obtained images it follows that the structure of the composite material is uniform with the presence of a large spread of distances between the fibers. The average diameter of carbon fibers T1000 was 5.42 microns.

A comparative analysis of the thin sections of the random zones of the microstructure of the composite material (fig. 4) indicates that the difference in the percentage of carbon fibers in the matrix between the samples cut from the equator is small (fig. 4, *a–c*), in all cases the microstructure is homogeneous. However, there is a significant difference between the sample cut from the equator (fig. 4, *b*) and the sample cut out in the flange zone (fig. 4, *d*). In the latter case, the microstructure of the sample cut out in the flange region (fig. 4, *d*) has a lower density of carbon fibers than on samples from the equator.

In the course of the study, the average values of the volume content of fibers in the composite tape were obtained for each sample, the results are shown in tab. 1.

The percentage of carbon fibers in the composite tape at the equator is 75.31 %, which corresponds to the optimal values according to the data of [3], which are in the range 65–83 %. However, in the case of MCHPT, where the thickness of the composite shell is variable, the percentage of carbon fibers varies with the transition from the equator to the flange region. Near the flange, the carbon fiber content was 61.33 %. The change in the percentage of fibers leads to a change in the characteristics of the mechanical properties of both the material and the shell as a whole.

Determination of porosity in the composite material of the MCHPT. To determine the porosity in the composite material, we used ESM at a 100-fold magnification of the image (fig. 5).
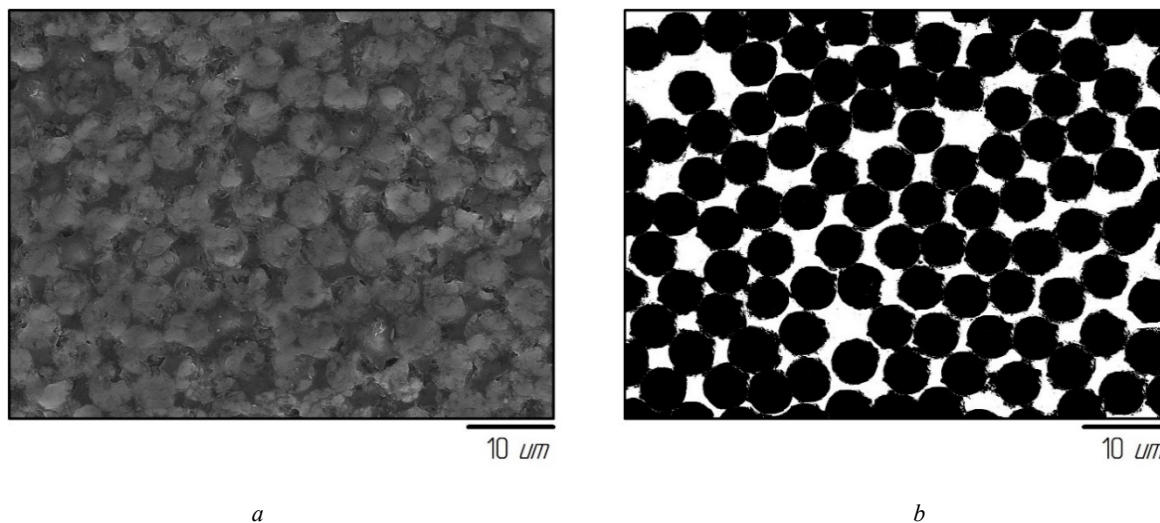

10 *um*	10 *um*
a	*b*

Fig. 3. The microstructure of the composite material at 2000 times magnification before (*a*) and after (*b*) computer processing

Рис. 3. Микроструктура композиционного материала при 2000-кратном увеличении до (*а*) и после (*б*) компьютерной обработки

Table 1

The values of the volume content of fiber in the matrix for the composite tape

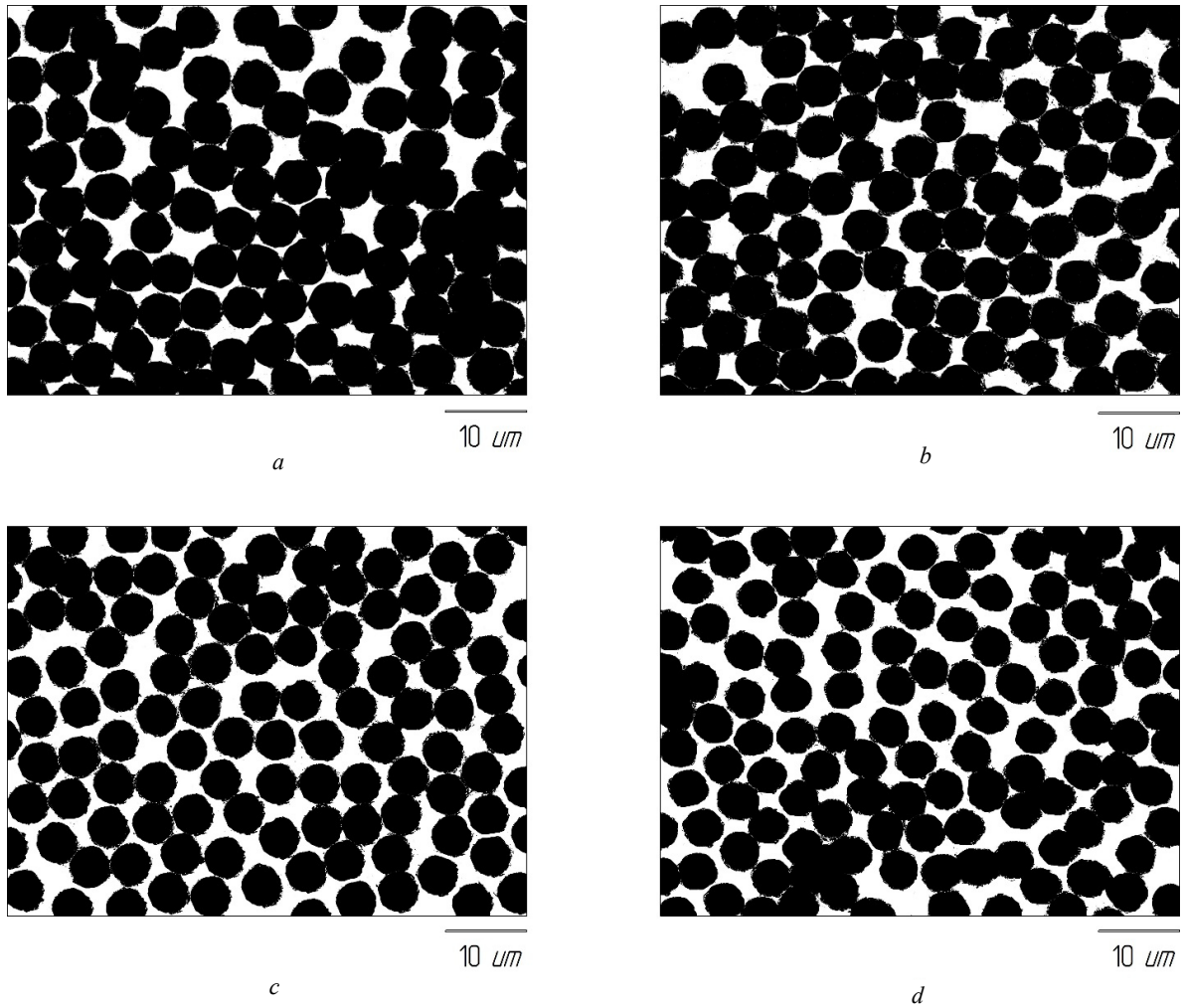| Sample	Average value of fiber content, %
Sample cut from the equator № 1	77.8
Sample cut from the equator № 2	75.39
Sample cut from the equator № 3	72.75
Sample cut from the flange № 4	61.33

Fig. 4. Comparative analysis of the microstructure of the composite material at 2000 times magnification:
a – first sample; *b* – second sample; *c* – third sample; *d* – fourth sample

Рис. 4. Сравнительный анализ микроструктуры композиционного материала при 2000-кратном увеличении:
а – первый образец; *б* – второй образец; *в* – третий образец; *г* – четвертый образец
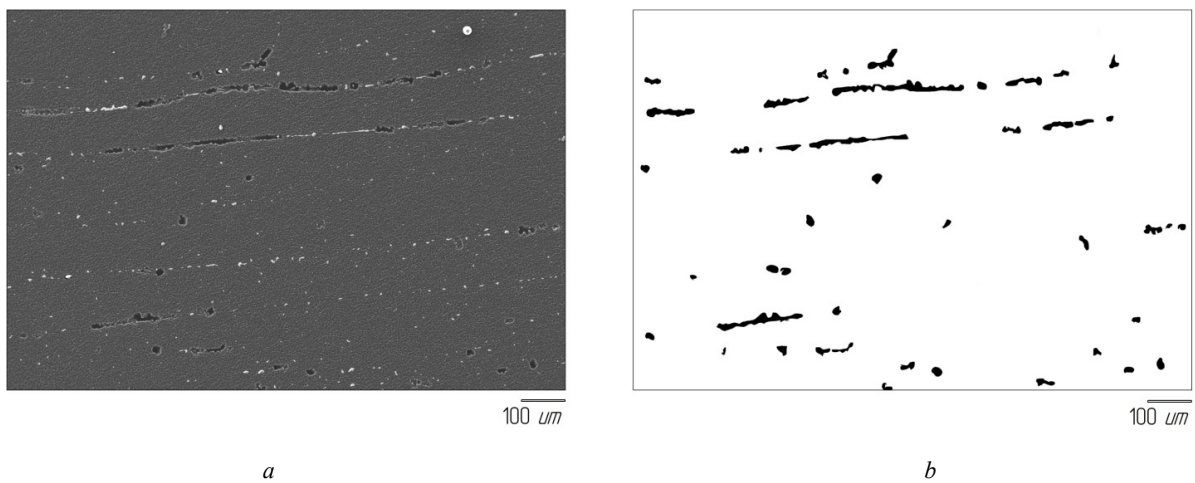

Fig. 5. The microstructure of the composite material at 100 times magnification before (*a*)
and after (*b*) computer processing

Рис. 5. Микроструктура композиционного материала при 100-кратном увеличении до (*a*)
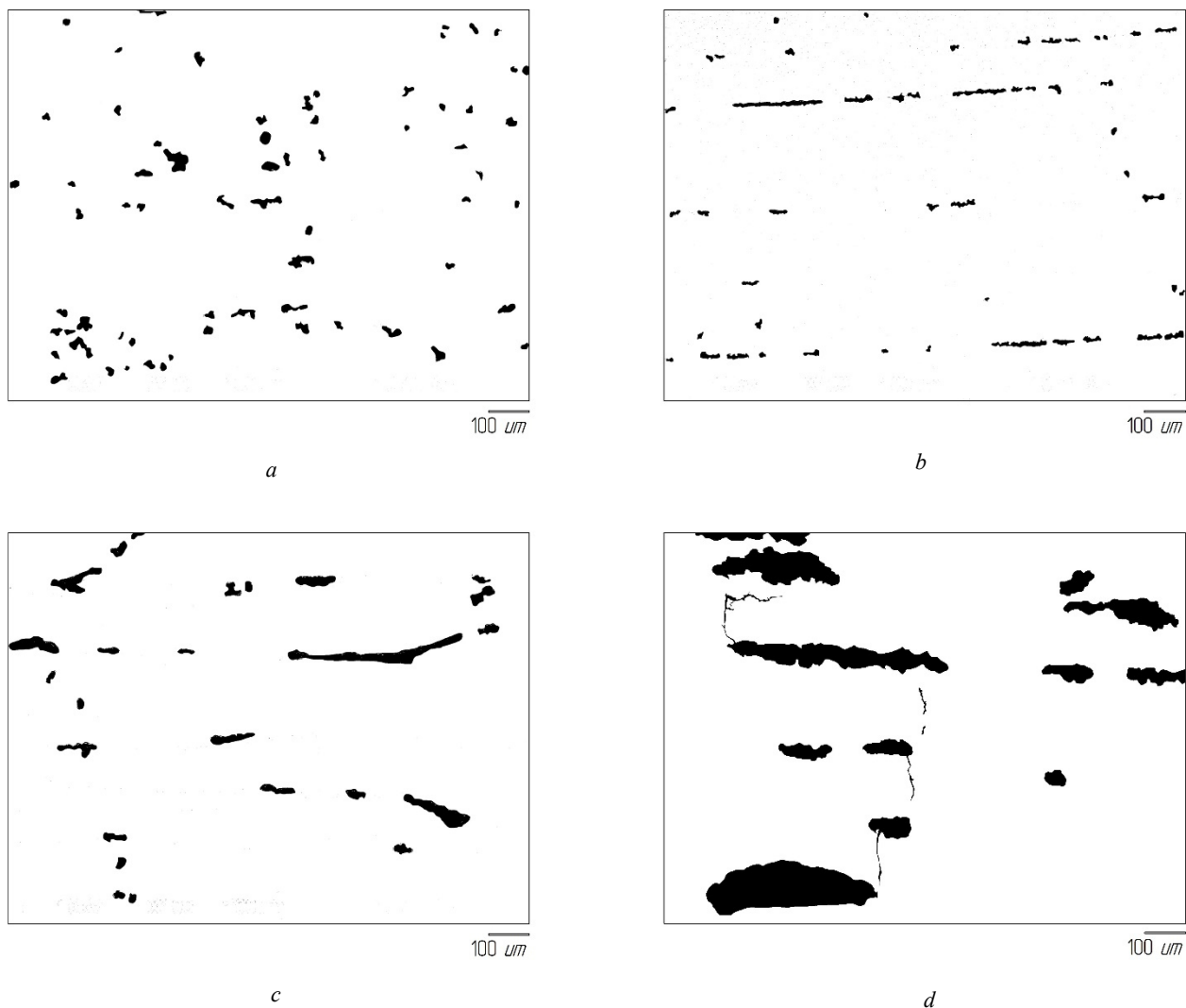и после (*б*) компьютерной обработки

Fig. 6. Comparative analysis of the microstructure of the composite material at 100 times magnification:
a – first sample; *b* – second sample; *c* – third sample; *d* – fourth sample

Рис. 6. Сравнительный анализ микроструктуры композиционного материала при 100-кратном увеличении:
а – первый образец; *б* – второй образец; *в* – третий образец; *г* –четвертый образец

Table 2

Characteristics of the porosity of the composite material of the MCHPT

Cut sample area	Pore occurrence, %	Average pore area, μm
Equator	2.32	726.58
Flange area	11.678	49038.63

A comparative analysis (fig. 6) showed that the microstructure of the laminate polymer composite material has a chaotic pore distribution. The pores on the equator (fig. 6, *a–c*) have small dimensions, and also frequent distribution over the surface. On the sample cut in the flange zone (fig. 6, *d*) there are critically large pores that create a stress concentration in the material. This is explained by the fact that during the winding of the composite tape in the flange area, the composite tape does not adhere well that leads to an excess of resin and the appearance of pores. This feature of the technological process is determined by the complexity of the elliptical shape of the liner.

During the research, the porosity characteristics of the composite material (percentage of porosity and average pore area) were determined. The results are shown in tab. 2.

Analysis of the structure of composite materials with different porosity shows that with the growth of the number of pores and their area, the strength characteristics of composite tapes and reinforcing fibers are reduced.

The most dangerous are elongated pores, the length of which exceeds the critical length of the fiber in the composite material. Such pores are stress concentrators and under external influences on the design or the occurrence of internal residual stresses in the material, they are the sources of the occurrence of microcracks.

Determination of the volume content of fibers in the composite material of the MCHPT. To determine the actual volume content of fibers in the matrix for a laminate polymer composite material, it is necessary to take into account the porosity of the material and the excess of epoxy resin according to the formula:

$$V_{\text{ф}} = V_{\text{кл}} \cdot (\text{П}_{\text{к}} + \text{И}_{\text{эс}}) \qquad (1)$$

where $V_{\text{кл}}$ is the volume content of fibers in the composite tape, %; $\text{П}_{\text{к}}$ is porosity of the composite material, %; $\text{И}_{\text{эс}}$ is epoxy resin excess.

Excess of epoxy occurs during the process of impregnating the composite tape with a binder between the layers. At the equator (sample No. 1–3), no epoxy resin excess is observed, however, an excess of epoxy resin between the layers is present in the flange zone (sample No. 4) (fig. 7). Due to the imperfection of the technological process of winding the composite tape onto the forming liner, this factor must be taken into account when calculating the actual volume content of fibers in the matrix.

Based on the photographs of the microstructures of the composite material cut out in the flange zone, the percentage of excess resin was determined as 7.44 %

The actual values of the volume content of the fibers for each sample were calculated using all the processed data obtained (tab. 1, 2) and are presented in tab. 3.

Calculation of the effective modulus of elasticity of the composite material of the MCHPT. The effective modulus of elasticity of a composite material is calculated using the "mixture rule" formula [3]:

$$E_{\text{к}} = E_{\text{н}} V_{\text{н}} + E_{\text{м}} V_{\text{м}}, \qquad (2)$$

where $E_{\text{н}}, E_{\text{м}}$ is the modulus of elasticity of the filler and matrix; $V_{\text{н}}, V_{\text{м}}$ is volumetric content of filler and matrix, %.

According to the formula (2), the strength of the composite material must increase proportionally to the volume content of the fiber in the matrix.

In addition to the "rules of mixtures", one can use the "polydisperse model of a medium with cylindrical inclusions" (CCA) to calculate the effective modulus of elasticity under uniaxial loading in the direction of the axis of a single compound cylinder [14]:

$$E_{\text{пд}} = V_{\text{н}} \cdot E_{\text{н}} + (1 - V_{\text{н}}) \cdot E_{\text{м}} + \frac{4(\nu_{\text{н}} - \nu_{\text{м}})^2 \cdot V_{\text{н}}(1 - V_{\text{н}})}{\dfrac{(1 - V_{\text{н}})}{K_{\text{н}}} + \dfrac{V_{\text{н}}}{K_{\text{м}}} + \dfrac{1}{G_{\text{м}}}}, \quad (3)$$

where, $\nu_{\text{н}}$, $\nu_{\text{м}}$ is Poisson's ratio of filler and matrix; $K_{\text{н}}$, $K_{\text{м}}$ is bulk modulus of elasticity of filler and matrix; $G_{\text{м}}$ is matrix shear modulus.

Formula (3) is the basic model in micromechanics, which was proposed by Hashin [15]. The peculiarity of this model is that additional characteristics of the mechanical properties of both the filler material and the matrix are taken into account.

100 *um*

a
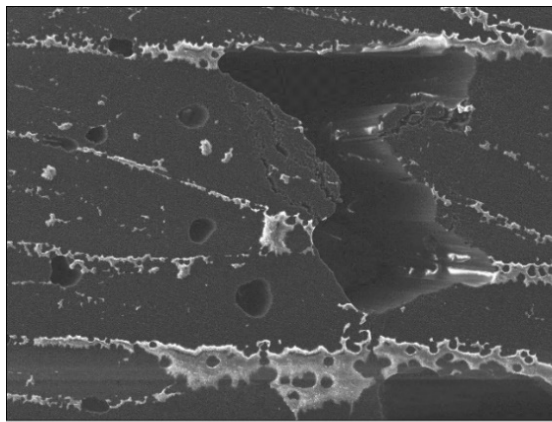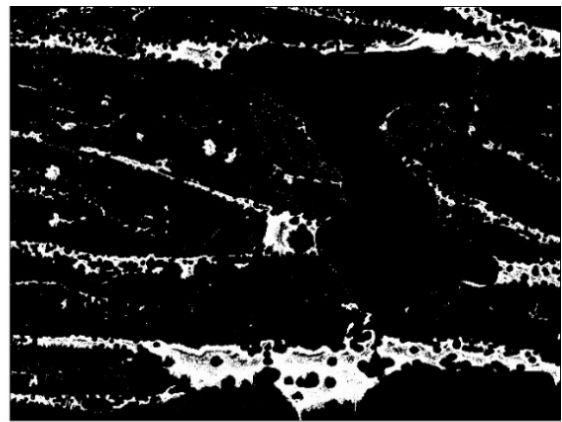
100 *um*

b

Fig. 7. The microstructure of the composite material near the flange at 100 times magnification before (*a*) and after (*b*) computer processing

Рис. 7. Микроструктура композиционного материала вблизи фланца при 100-кратном увеличении до (*а*) и после (*б*) компьютерной обработки

Table 3

The values of the volume content of fibers in the matrix for the composite material of the MCHPT

Sample	The actual volume content of fibers in the matrix, %
Sample cut from the equator № 1	76
Sample cut from the equator № 2	73.64
Sample cut from the equator № 3	71.06
Sample cut from the flange № 4	49.6

Table 4

Characteristics of the mechanical properties of carbon fiber T1000 and epoxy resin ED-I

Characteristic	$E_{\text{н}}$	$E_{\text{м}}$	$G_{\text{м}}$	$K_{\text{н}}$	$K_{\text{м}}$	$\nu_{\text{н}}$	$\nu_{\text{м}}$
Value	290 hPa	3 hPa	1.13 hPa	219.69 hPa	2.7 hPa	0.28	0.32

Table 5

The values of the effective modulus of elasticity of the composite material of the MCHPT

Sample	Effective modulus of elasticity, hPa
Sample cut from the equator № 1	221.12
Sample cut from the equator № 2	214.34
Sample cut from the equator № 3	206.94
Sample cut from the flange № 4	144.63

The mechanical characteristics of the composite material MCHPT were taken from the technical documentation of the product and are presented in tab. 4.

According to formulas (2) and (3), elastic moduli of the composite material for each sample were determined. The elastic moduli determined by the "polydisperse model" are consistent with the modules determined by the "rule of mixtures". Taking into account additional parameters of the model gives insignificant differences. In this regard, the results presented only for "polydisperse" model are reflected in table 5.

The modulus of elasticity of the composite material MCHPT near the flange is 32 % less than the modulus of elasticity of the composite material from the equator. However, the "mixture rule" and "polydisperse model" for composites with unidirectional fibers are based on the assumption that the strength of the composite depends only on the strength and the ratio of the components, and also that the bond at the interfaces is ideal. However, these equations are not capable of giving a reliable estimate of the strength of a laminated composite material. The stiffness and strength of a laminated polymeric composite are mainly determined by the outer layers and the contribution of each layer [16]. For this reason, the obtained effective modulus of elasticity may not correspond to the experimental data presented in the study [17].

Conclusion. The results of the study made it possible to give a comprehensive evaluation of the quality of the layered polymeric composite material of MCHPT using parameters characterizing the structure of the material.

Based on the results obtained, the volume content of carbon fibers (taking into account porosity) in the composite material of the MCHPT at the equator is 73.56 %, which corresponds to the optimal values. However, near the flange, the volume content of carbon fibers is 49.6 that is significantly lower than the optimal values.

Despite the overestimation of the effective modulus of elasticity of the composite material of the MCHPT, the "mixture rule" and the "polydisperse model" are useful for approximate calculations. From tab. 5, it can be concluded that the effective modulus of elasticity in the equatorial zone and near the flange has a large difference in values. In this regard, in the design calculations of the stress-strain state of the MCHPT, it is necessary to take into account the heterogeneity of the mechanical properties of the composite shell.

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