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EVALUATION OF INFLUENCE OF METHOD FOR PREPARING ON MICROPOROSITY OF SINTERED POLYTETRAFLUOROETHYLENE-BASED COMPOSITIONS

Abstract. The aim of the work is to assess the influence of the method for preparing on the porosity of sintered polytetrafluoroethylene blanks as well as PTFE mixtures with carbon fiber and additives of powdered graphite. The article provides a comparative analysis of the method for preparing influence on the microporosity of sintered blanks made of filled and unfilled polytetrafluoroethylene. Microporosity has been determined through the comparison of the actual and theoretical (for a non-porous material) density of blanks, calculated by the methods of structural mechanics of composites. The studies made it possible to establish that the porosity of the unfilled polytetrafluoroethylene blanks pressed at a pressure of 70...80 MPa stands at 1.3 to 5.9 %. An exception was a specimen obtained by sintering in a jig, which has reached apparent density 2200 kg/m³ equal to theoretical density. It has been established, that the porosity of blanks obtained from polytetrafluoroethylene filled with shredded carbon fiber and powdered graphite stood at 0.4 to 3.9 %. An exception was a specimen with a high mass content of filler (40 %), in which the porosity was 16 %. It has been ascertained that sintering in a constrained state helps to reduce the residual microporosity for both the filled and unfilled polytetrafluoroethylene. This shows the technical efficiency of sintering in a constrained state, despite the increased labor intensity of the manufacturing process and the sophistication of technical equipment.

Keywords: polytetrafluoroethylene, raw pressing composition, carbon fiber, graphite, density, porosity

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ОЦЕНКА ВЛИЯНИЯ СПОСОБА ПОДГОТОВКИ НА МИКРОПОРИСТОСТЬ СПЕЧЕННЫХ ЗАГОТОВОК ИЗ КОМПОЗИЦИЙ НА ОСНОВЕ ПОЛИТЕТРАФТОРЭТИЛЕНА

Аннотация. Представлены результаты работы, целью которой являлась оценка влияния способа подготовки на пористость формованных заготовок из порошкообразного политетрафторэтилена, а также его смесей с углеродным волокном и добавками пылевидного графита. Для достижения поставленной цели был выполнен сравнительный анализ влияния способа подготовки на микропористость спеченных заготовок из наполненного и ненаполненного пресс-материала на основе политетрафторэтилена. Методология определения микропористости заключалась в расчете соотношения фактической плотности заготовок и теоретической для беспористого материала, определенной методами структурной механики композитов. Результаты выполненных исследований показали, что заготовки из ненаполненного фторопласта, спрессованные при давлении 70...80 МПа, как правило, имеют пористость от 1,3 до 5,9 %. Исключение составляет образец, полученный спеканием в условиях всестороннего сжатия, у которого была достигнута нулевая пористость. Пористость заготовок, полученных из фторопласта, наполненного измельченным углеродным волокном и пылевидным графитом, составляет в абсолютном большинстве случаев от 0,4 до 3,9 %. Исключение представляет образец с содержанием наполнителя 40 мас.%, у которого пористость равна 16 %. Как для исходного, так и для наполненного политетрафторэтилена спекание в условиях всестороннего сжатия во всех опытах способствовало уменьшению остаточной микропористости. Это свидетельствует о технической эффективности такого метода получения изделий, несмотря на повышение трудоемкости процесса и усложнение средств технического оснащения.

Ключевые слова: политетрафторэтилен, пресс-композиция, углеродное волокно, графит, плотность, пористость

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Introduction. Composite materials based on polytetrafluoroethylene (PTFE) have a complex of unique properties including abnormally low coefficient of friction when working without lubrication, in vacuum and at cryogenic temperatures as well as high chemical resistance to the action of reagents and biofluids, and they are widely used in various industries. Products of PTFE are used in friction units in mechanical engineering [1–6], including chemical machines and units [7, 8], in compressor technique [9], electrical engineering [10] and medicine [11]. Products from PTFE-based compositions are manufactured by pressing blanks from a pre-prepared mixture of components (raw pressing composition) followed by sintering [12].

Despite the progress achieved in the field of creating fluorine composites, many phenomena occurring during pressing and sintering processes are insufficiently studied [13–16]. In particular, the scientific and technical literature does not say enough about the issues of possible existence of residual microporosity in the blanks. The existence of such microporosity in fluorine composites was experimentally established in a number of papers [17–20]. However, the patterns of its formation have not been adequately studied. It is known that the process of pressing powder materials includes several successive stages [21, 22]: elastic deformation of filler particles and polymer matrix, emergence of a plastic state area (stage of constrained elastic-plastic deformation), shift of this area to the surface of particles (free plastic flow with an intensive change in the porosity and volume of the pressed material, an increase of its density). At the final stage of the deformation process the deformable material “blocks” the remaining pores and thus hinders the release of gaseous products contained in them, which is the reason for the preservation of the residual microporosity of the pressed material after removing the load. Subsequently, while sintering blanks, this microporosity can not only persist, but even increases, since sealed air remaining in the micropores expands in volume with a rise of temperature. This final stage of blanks deformation has not been adequately studied.

The aim of the work is to assess the influence of the method for preparing on the porosity of sintered polytetrafluoroethylene blanks as well as PTFE mixtures with carbon fiber and additives of powdered graphite.

Methodology for theoretical and experimental research. Experimental studies were carried out in two phases. At the first phase, the influence of the methods for preparing blanks of unfilled PTFE on their density and microporosity was assessed, and at the second phase a similar series of experiments on composites containing carbon fibers and powdered graphite was carried out. As the object of research composites based on PTFE of brand PN-90 according to State Branch Standard 10 007-80 (density $\rho_m = 2200 \text{ kg/m}^3$) were used. The following materials were used as fillers: carbon fiber (CF) of brand LO-1-12N produced by JSC SvetlogorskKhimvolokno (density $\rho_f = 1500 \text{ kg/m}^3$) and powdered graphite (PG) according to Technical Conditions 48-4802-20-90 (density $\rho_g = 2250 \text{ kg/m}^3$).

The theoretical density of non-porous composite ρ_{klim} was determined by the methods of structural mechanics under the condition of conservation the mass of the system. The mass of gaseous products inside the pores was neglected while evaluation the porosity of the specimens due to its insignificance in comparison with the mass of the resulting products.

The schemes of the specimen preparation are shown in Table 1.

Table 1. Compound and manufacturing technology of specimens from PTFE-based composites

Compo- site symbol	Compound of the composite	Technology particularities
K1	PTFE – 83 %; CF – 16.5 %; PG – 0.5 %	Tribostatic spray deposition of ultrafine PTFE on carbon fiber tape followed by exposure at 340 °C for 20 min; normal sintering mode
K2	PTFE – 70 %; CF – 29.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; normal sintering mode
K3	PTFE – 75 %; CF – 24.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; normal sintering mode
K4	PTFE – 70 %; CF – 29.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; sintering in the jig
K5	PTFE – 75 %; CF – 24.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; sintering in the jig
K6	PTFE – 70 %; CF – 29.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; sintering in the jig; compression of the blank in the pressing mold after sintering
K7	PTFE – 60 %; CF – 39.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; sintering in the jig
K8	PTFE – 75 %; CF – 24.5 %; PG – 0.5 %	Mechanical activation of the compound by rolling; sintering in the jig; compression of the blank in the pressing mold after sintering

The technological operations shown in Table 1 were carried out in accordance with technical regulation, which embraced at the initial stage the preparation of raw materials. This readying included shredding a carbon fiber tape to obtain fragments of carbon fibers of the required length, as well as mechanical activation of PTFE in a paddle mixer with paddle rotation frequency of not more than 1500 s^{-1} . Further, the prepared fillers (CF and PG) were loaded into the paddle mixer, which already contained the mechanically activated PTFE.

Upon manufacturing of the composite, conventionally marked in Table 1 as “K1”, the carbon tape fiber was coated with the ultrafine PTFE by spraying it from a tribostatic gun. Afterward the powder-coated carbon fiber tape was kept at $340 \text{ }^\circ\text{C}$ for 20 min, and then shredded.

In order to reduce the porosity of the composite and increase the efficiency of the sintering process, the compound was mechanically activated by rolling before pressing blanks (in Table 1 – “mechanical activation of the compound by rolling”, specimens K2–K8). The prepared portion of the composite material was passed between two rotating rolls at roll-speed-ratio of 1 : 1.25. Thus, the brittle film of the composite material was obtained, which then was grinded in a four-paddle mixer. The compound was dried in drying ovens with forced air circulation in trays at a temperature of $150 \pm 5 \text{ }^\circ\text{C}$ for 4 h.

The blanks pressing process was carried out in steel pressing molds with a hydraulic press in all cases. Weighed dosing was used to obtain the exact height of the blanks. After drying in the oven and weighing the compound was freely filled in the pressing mold and carefully leveled with a soft-metal plate in order to achieve uniform density.

Three stops were made from the moment of initial compression of the compound until the moment when the manometer begins to register the final target pressure. After each stop, a 5 s exposure was carried out. Upon reaching the final target pressure, the exposure was carried out for 3 min. The gauge pressure was adjusted to achieve a specific compression pressure of 70–80 MPa.

The blanks were sintered in electric furnaces with a rotating hearth and forced air circulation in a free state as shown in [19], or under conditions of all-round compression using jigs [17]. The blanks were loaded into the furnace at a temperature of 20 to $50 \text{ }^\circ\text{C}$. The heat treatment of the blanks was carried out according to the regime providing for heating to $360 \text{ }^\circ\text{C}$ at a rate of 0.5 deg/min and exposure upon reaching $150 \text{ }^\circ\text{C}$ and $312 \text{ }^\circ\text{C}$ for 3 h. Sintering was performed at a temperature of $360 \text{ }^\circ\text{C}$. Further, the blank was cooled at a rate of 0.5 deg/min to a temperature of $\leq 50 \text{ }^\circ\text{C}$ with exposures for 3 h at a temperature of $325 \text{ }^\circ\text{C}$ and $150 \text{ }^\circ\text{C}$.

Specimens K6 and K8 after the heat treatment were exposed under the pressure level of 70–80 MPa in a pressing mold with specified geometric dimensions in accordance with the approach described in [23].

The apparent density of the composite after sintering the blanks was assessed according to State Branch Standard 15139-69 by the method of measurement and weighing. Specimens were rods with geometric dimensions $\varnothing 23 \times 70$. The mass of the specimens fluctuated from 51 to 55 g, and the volume was from 27.75 to 29.64 cm^3 .

The experimental data were processed using standard statistical methods. The arithmetic mean values of the indicators, the standard deviation were defined. The confidence interval according to a Student’s T-test was determined at a confidence level of 0.95. The number of tests in experiments was at least 5 for each kind of material. The experimental data were processed on a computer using the standard Microsoft Office 11 software.

The theoretical (limit) density of a non-porous composite was calculated by the formula

$$\rho_{kt} = \frac{\rho_m}{c_m + c_f \frac{\rho_m}{\rho_f} + c_g \frac{\rho_m}{\rho_g}}, \quad (1)$$

where c_m , c_f , c_g – the mass content of the matrix, carbon fibers and graphite respectively in wt.%; ρ_{kt} , ρ_m , ρ_f , ρ_g – the densities of the ideal non-porous composite, matrix, carbon fibers and graphite respectively.

The porosity of the composite can be described by the formula

$$\theta_p = 1 - \frac{V_{up}}{V_k} = 1 - \frac{m_k}{\rho_{kt}} \cdot \frac{\rho}{m_k} = 1 - \frac{\rho}{\rho_{kt}}, \quad (2)$$

where θ_p – the porosity of composite; V_{up} – the volume of the non-porous (monolithic) part of composite; V_k – the volume of the composite; m_k – the mass of the ideal non-porous composite; ρ – actual density of the composite.

Formula (2) is the alternative to State Branch Standard P 56679-2015 and makes it possible to calculate the porosity of blanks and products. It can be used under the conditions of the known composite compound, theoretical and actual density. In doing so, the theoretical density is calculated via formula (1) and actual density should be measured experimentally.

Research results and discussion. The Table 2 shows the effect of various technologies of specimen preparation on the density and microporosity of unfilled PTFE.

Mechanical activation of powdered PTFE by rolling and sintering in a jig were carried out according to the technology similar to the mentioned in the Table 1 above.

Table 2. Actual values of density and porosity in unfilled PTFE

Technology particularities	Actual density, kg/m ³	Relative density, %	Porosity, %
Mechanical activation of powdered PTFE by rolling + quench hardening	2071	94.1	5.9
Normal sintering mode	2113	96.0	4.0
Quench hardening	2171	98.7	1.3
Mechanical activation of powdered PTFE by rolling + normal sintering mode	2137	97.1	2.9
Sintering in the jig	2200	100.0	0.0
Mechanical activation of powdered PTFE by rolling + sintering in the jig	2160	98.2	1.8

Quench hardening of unfilled PTFE was carried out in water at 20 °C in order to improve the physical and mechanical properties due to reduce in the degree of material crystallinity. The obtained value of the actual density was 2171 kg/m³ and exceeded the level considered normal for the hardened PTFE (2150 kg/m³) [24]. The most likely cause for the increased density was that the degree of crystallinity of the specimen was above 50 % due to the relatively considerable thickness of the specimen (23 mm). In this case the low thermal conductivity of PTFE has led to the fact that hardening had not occurred in the entire volume of the specimen.

Analysis of the data in Table 2 shows that the porosity of the blanks from unfilled PTFE fluctuated from 1.3 to 5.9 %. An exception was a specimen manufactured by sintering in a jig, which has reached the zero porosity (density 2200 kg/m³). This density value indicates both the monolithic state and a high degree of crystallinity of the blanks' material. In combination, it allows expecting a relatively high hardness and resistance to loads of the product, which is in line with the test results.

The Table 3 shows the results of calculating the theoretical (limit) and relative density, as well as porosity of PTFE filled with carbon fibers and powdered graphite in dependence of technology particularities.

The analysis of the data in Table 3 shows that the porosity of the specimens of PTFE filled with carbon fiber and powdered graphite varied in the great majority of cases from 0.4 to 3.9 %. An exception was a specimen K7, which had the 16 % porosity rate. However, this should not be considered as an experimental error, since similar porosity parameters ($\theta_p = 15.4–15.9$ %) were also recorded by other researchers during free sintering of composites [22]. Apparently, such a large gap in the value of the porosity between the specimen K7 and the rest of the blanks could be explained by the existence of a certain limit content value of fillers at which PTFE of PN-90 brand can perform the function of monolithic

Table 3. The results of calculating the theoretical (limiting) density, as well as the relative density and porosity of composites

Composite symbol	Density of specimens			Calculated porosity, %
	theoretical for non-porous material, kg/m ³	actual, kg/m ³	relative, %	
K1	1962.70	1887	96.1	3.9
K2	1861.93	1814	97.4	2.6
K3	1899.44	1858	97.8	2.2
K4	1861.93	1801	96.7	3.3
K5	1899.44	1892	99.6	0.4
K6	1861.93	1821	97.8	2.2
K7	1971.19	1655	84.0	16.0
K8	1899.44	1892	99.6	0.4

matrix in a polymer composite material. When the content of the filler exceeds the 30–40 wt.%, it becomes extremely difficult to avoid the formation of microclusters of the fibrous modifier, which cannot be destroyed by any of the existing technological methods. Further the microclusters become a zone of accumulation of air microinclusions, which increase in volume with a rise in temperature while sintering, and cause the increased porosity of products. The influence of the mass content of the filler on the possibility of forming a non-porous blank from a PTFE-based composite is confirmed by the fact that the calculated porosity of the blanks with 25 wt.% filler turned out to be significantly lower than the calculated porosity of the blanks with 30 wt.% filler, while manufacturing technology was identical ($K_3 < K_2$, $K_5 < K_4$, $K_8 < K_6$). Nevertheless, the relatively high porosity of specimen K1 (filler content 17 wt.%), as well as unfilled PTFE specimens (see Table 2), indicates that up to 30 wt.% the technology is crucial for the formation of a non-porous structure of blanks made of PTFE-based composites.

Applying such techniques as mechanical activation of the compound by rolling, sintering in a jig and compression of a blank in the pressing mold after sintering makes it possible to reduce the porosity of the products of filled PTFE even below the products of unfilled PTFE.

The result can be considered to be obtained due to a decrease of size and quantity of pores, which, in the authors' opinion, were the result of significant differences in the parameters of electrophysical, thermophysical, mass and dimensional characteristics of the particles in the pressing compound. Sintering in the jig creates the conditions for the elimination of microcavities due to the peculiarities of the thermo-physical characteristics of PTFE particles and CF fragments. Thermal expansion of the components in a jig causes strain stresses, which then presumably lead to the generation of interfacial layer PTFE–CF with lower defectiveness of structure. The absolute majority of CF fragments become to be connected by polymer interlayers, which increase the strengthening effect of the filler due to the rise of the proportion of the mechanical component of the adhesive force at the interfacial layer [23].

Application of the developed techniques allows increasing the parameters of stress-strain and tribotechnical characteristics of PTFE-based composites 1.5–2 times in comparison with current technical regulation. Testing of seals made of PTFE-based composites in compressor equipment at chemical enterprises (e.g., JSC “Grodno Azot”) have shown that the application of technique of compression of the blank in the pressing mold after sintering can increase the service life of seals at least by 2 times. And piston rings made by sintering in the jig worked 3 times more than similar products obtained by normal sintering mode (e.g., at JSC “SMNPO – Engineering” compressors).

Conclusion. The effect of the method for preparing on the microporosity of sintered blanks of filled and unfilled polytetrafluoroethylene was evaluated by the methods of structural mechanics of composites and experimentally. Microporosity was calculated as the ratio of the actual measured density to the theoretical density of a non-porous material, evaluated by the methods of structural mechanics of composites. The research findings allowed claiming that the porosity of the pressed at a pressure of 70–80 MPa unfilled PTFE blanks was from 1.3 to 5.9 %. An exception was a specimen obtained by sintering in a jig, which has reached zero porosity (density 2200 kg/m³). The porosity of blanks from PTFE filled with carbon fiber and powdered graphite was from 0.4 to 3.9 %. An exception was a specimen with a high mass content of carbon fibers (compound: PTFE – 60 %; CF – 39.5 %; PG – 0.5 %), in which the porosity was 16 %. Sintering in a constrained state (in a jig) in all experiments led to the reduction of the residual microporosity for both the unfilled and filled PTFE. That proves the technical efficiency of this technique, despite the increased labor intensity of the process and the sophistication of technical equipment.

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