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Kinetics of cold compaction of polytetrafluoroethylene-based composite material taking into account structural factors in constant force modes on press plunger or its constant speed

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Abstract: The article discusses the results of mathematical modeling of the one-sided pressing technological process of powder fluoropolymer materials. The peculiarity of the theoretical description is the consideration of rheodynamics, structuring and kinetics of compaction of the compressed medium. An important point of such a description is the choice of rheological equations. In what follows, it is assumed that the compaction of the material occurs according to the mechanism of viscous flow of the mass into pores (according to the theory of Ya.I. Frenkel). The rheological properties of such a medium, i.e. the ability to deform and flow, are determined by the properties of the solid phase, the presence and degree of porosity. Two variants of the technological process of pressing are considered depending on the externally specified conditions for the movement of the press plunger: modes of a constant specified force or its constant speed. The analysis of numerical calculations for each of these modes made it possible to identify their fundamental features. It was found that in the mode of a specified force, progressive autobraking of the compaction process occurs over time. It is shown that for pressing materials in both modes it is necessary to select such parameters when the time of structural transformations is longer than the compaction time. The conducted analysis allowed to develop specific recommendations for forecasting rational modes of one-sided pressing of powder materials.

Keywords: fluoropolymers; polytetrafluoroethylene; structural model; cold pressing; rheodynamics; structurization.

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Кинетика холодного уплотнения композиционного материала на основе политетрафторэтилена с учетом структурных факторов в режимах постоянного усилия на плунжере пресса или его постоянной скорости

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Аннотация: Обсуждаются результаты математического моделирования технологического процесса одностороннего прессования порошковых фторполимерных материалов. Особенностью теоретического описания является учет реодинамики, структурирования и кинетики уплотнения сжимаемой среды. Важный моментом такого описания – выбор реологических уравнений. В дальнейшем предполагается, что уплотнение материала происходит по механизму вязкого течения массы в поры (согласно теории Я. И. Френкеля). Реологические свойства такой среды, то есть способность к деформированию и течению, определяются свойствами твердой фазы, наличием и степенью пористости. Рассмотрены два варианта технологического процесса прессования в зависимости от задаваемых извне условий на перемещение плунжера пресса: режимы постоянного заданного усилия или его постоянной скорости. Анализ численных расчетов для каждого из этих режимов позволил выявить их принципиальные особенности. Установлено, что в режиме заданного усилия происходит прогрессивное автоторможение процесса уплотнения во времени. Показано, что для прессования материалов в обоих режимах следует выбирать такие параметры, когда время структурных превращений больше времени уплотнения. Проведенный анализ позволил выработать конкретные рекомендации прогноза рациональных режимов одностороннего прессования порошковых материалов.

Ключевые слова: фторполимеры; политетрафторэтилен; структурная модель; холодное прессование; реодинамика; структуризация.

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1. Introduction

It is known that deformation has a strong effect on the structure of the forming polymer and on the kinetics of viscosity changes during technological processes. Despite the successes achieved and a large number of published works in this area, there are still phenomena and dependencies which nature is still unclear. This is quite understandable if we take into account the fact that problems of structural-deformational dependencies cover a wide range of processes, phenomena and objects in the chemistry and mechanics of polymers [1, 2]. During shear deformation of fluid structured systems, a phenomenon of viscosity superanomalies, named so by the founder of domestic polymer rheology G.V. Vinogradov, is realized [3]. This phenomenon is associated with a decrease in resistance to deformation with an increase in the deformation rate. In this case, the rheological curve in the coordinates of stress from the shear rate has an N-shaped form, and one of the branches of this curve has a negative slope (negative differential viscosity). It should be noted that under conditions of super-anomaly of viscosity, a sudden transition through the critical value is possible, which leads to the fact that the deformation mode with an almost intact structure and high viscosity abruptly changes to the deformation mode with an extremely destroyed structure and low viscosity.

Previously, a theoretical analysis of the process of one-sided pressing under conditions of a constant speed on the press plunger and constant pressure of powder composite polymer materials based on polytetrafluoroethylene (PTFE) with small additives (less than 5 %), such as silicon dioxide SiO_2, kaolinite $Al_4[Si_4O_{10}](OH)_8$, carbon nanotubes, carbon fiber was carried out. New visual ideas about the kinetics of compaction of powder materials based on polytetrafluoroethylene under conditions of constant pressure and speed of the press plunger were established [4].

This paper presents a mathematical model of the process of cold pressing of a viscous structured material. Previously developed rheodynamic models of solid-phase pressing of materials [4, 5] are supplemented by taking into account the dependence of viscosity on the structural parameter.

It is assumed that under the action of external loads the structure of the material can be destroyed. The processes of structural transformations, namely, the destruction and restoration of the structure, are similar to the process of a chemical reaction occurring in the forward and reverse directions [3, 6, 7]. But rheodynamics was not taken into account in the theoretical description of these processes.

The structural approach allows us to analyze the dynamics of the deformation processes of structured systems and explain the nature of various critical (threshold) phenomena and oscillatory modes [7, 8]. This approach made it possible to analyze the dynamics of the deformation process of structured systems. Based on a numerical study of the problem posed for the process parameters corresponding to solid-phase pressing of fluoropolymers, the possibility of various deformation modes was theoretically shown.

2. Process model and problem statement

The compaction of a viscous porous material in a cylindrical chamber limited from above by a moving piston should be considered. The axis of symmetry of the work piece is taken as the *z* axis, the positive direction of which is opposite to the direction of piston movement.

The rheodynamic model of the process, which does not take into account structural transformations, is presented in [4]. The formulation of the problem taking into account structural transformations includes equations, equilibrium (1), continuity (2), rheological relationships (3), (4), and a kinetic equation describing the change in structure (5):

$$\frac{\partial \sigma_{zz}}{\partial z} = 0; \qquad (1)$$

$$\frac{\partial \rho}{\partial t} + \frac{\partial (\rho V)}{\partial z} = 0; \qquad (2)$$

$$\sigma_{zz} = \left(\frac{4}{3}\mu + \xi\right)\frac{\partial V}{\partial z}; \qquad (3)$$

$$\sigma_{rr} = \sigma_{\theta\theta} = \left(-\frac{2}{3}\mu + \xi\right)\frac{\partial V}{\partial z}; \qquad (4)$$

$$\frac{\partial (a\rho)}{\partial t} + \frac{\partial (a\rho V)}{\partial z} = \varphi\left(a\sigma_{zz}\right), \qquad (5)$$

where $\sigma_{zz}, \sigma_{rr}, \sigma_{\theta\theta}$ are axial, radial and tangential stresses; ρ is relative density of the material; μ, ξ is shear and bulk viscosity of the material; V is flow rate of the material; ρ is density of the incompressible base of the material, a is a degree of structural changes. Due to the high viscosity of fluoropolymers, the Reynolds criterion is small [8, 9], therefore, for cold pressing of fluoropolymers, the equilibrium equation (1) can be used instead of the equation of motion.

To study the process of structural transformations, we use a model of the flow of a two-component liquid taking into account the kinetics of the mutual transformation of structural units [8].

Let the rheological system consist of structures of types A and B, mutually transforming into each other $A \underset{\leftarrow}{\overset{\rightarrow}{—}} B$ under the action of an applied mechanical field with their concentration in the volume a and $b = 1 - a$. In this case, the kinetic equation for the change in structure is written as (5). We assume that the processes of restoration and destruction of the structure are activation processes, the rates of destruction and restoration of structures can be written as:

$$k_1 = k_{10} \exp\left(-\frac{U_1 - E_1}{RT}\right);$$

$$k_2 = k_{20}\left(-\frac{U_1 + E_1}{RT}\right),$$

where T is the sample temperature; U_1 is the activation energy of viscous flow; R is a universal gas constant; E_1 is the effective activation energy; k_{10}, k_{20} are constants of destruction and restoration of the structure, respectively. For cold pressing, the temperature does not change during the process and is equal to the ambient temperature (293 K).

Then the total rate of structural transformations:

$$\varphi(a\sigma_{zz}) = -k_1 a + k_2(1-a).$$

Under the influence of a mechanical field, deformation of bonds in the structure being destroyed occurs, depending on the magnitude of the stress, and the orientation of randomly directed molecular-kinetic units, depending on the velocity gradient. In this case, the effective activation energy $E_1 = p_1\sigma_{zz}$, where p_1 is the constant of the destruction intensity. Due to the high viscosity of fluoropolymers (10^8–$4\cdot10^9$ Pa·s), orientational rotation is difficult and the effective activation energy decreases only under the action of compressive (or tensile) stress (this applies to the mechanical destruction of polymers).

In this paper, the generalized Newton model is used to describe the viscoelastic behavior of the material:

$$\sigma_{zz} = \left(\frac{4}{3}\mu + \xi\right)\frac{\partial V}{\partial z},$$

where $\mu = \mu(a) = \mu_0 \exp(ka)$ is the viscosity depending on the degree of structural transformations a (the concentration of intermolecular crosslinks).

Usually, different deformation modes are set in the experiment: a mode with constant force (a constant pressure P is set on the piston) and a mode with a constant deformation rate (a constant piston speed is set): $\sigma_{zz}\big|_{z=H(t)} = -P$, $V_{zz}\big|_{z=H(t)} = V$. In this paper, both deformation modes are considered.

The problem is characterized by the presence of a moving boundary: the upper boundary of the solution region ($Z = H$), corresponding to the press plunger

$$\frac{dH}{dt} = -V_H.$$

The problem has two viscosity coefficients – shear (μ) and volume (ξ), depending on the density and structural parameter (a):

$$\mu(\rho, T, a) = \mu_0\rho^m \exp(ka);$$

$$\xi(\rho, T) = \frac{4}{3}\mu(\rho, T, a)\rho/(1-\rho) = \frac{4}{3}\mu_0\rho^{m+1}/(1-\rho)\exp(ka).$$

At the initial moment of time, the density distribution by the pressing height is given $\rho(z, 0) = \rho_0(z) = \rho_0 + (\rho_m - \rho_0)z/H_0$ and the initial distribution of the structure by sample $a(z, 0) = a_0(z)$.

Boundary conditions:

$z = 0$: $V = 0$;

$z = H_0$: $\sigma_{zz} = -P$ (for the case of a given force on the press plunger);

$z = H_0$: $V_n = V_0$ (for the case of a given speed on the press plunger).

For the numerical solution of the problem, Lagrangian coordinates associated with the medium for stopping the upper moving boundary of the sample were used. Lagrangian coordinates (q; t) have the following meaning: $t_L = t$ is real time, mass coordinate q has the meaning of the relative mass between the lower boundary of the mold and the upper moving boundary z:

$$q = \int_0^z \rho(z,\, t)\,dz,$$

$q_0 = \int_0^{H_0} \rho(z, 0)dz$ is the initial coordinate, which is the total mass of the sample, expressed in linear units. The problem statement in Lagrange coordinates has the following form:

$$\frac{\partial \sigma_{zz}}{\partial q} = 0;$$

$$\frac{\partial \rho}{\partial q} + \rho^2 \frac{\partial (V)}{\partial q} = 0;$$

$$\sigma_{zz} = \left(\frac{4}{3}\mu + \xi\right)\rho \frac{\partial V}{\partial q};$$

$$\sigma_{rr} = \sigma_{\theta\theta} = \left(-\frac{2}{3}\mu + \xi\right)\rho \frac{\partial V}{\partial q};$$

$$\frac{\partial (a)}{\partial t} = \varphi(a, \sigma_{zz})/\rho_1 \rho\ .$$

Boundary conditions:

$q = 0$: $V = 0$;

$q = q_0$: $\sigma_{zz} = -P$ (for the case of a given force on the press plunger);

$q = q_0$: $V_n = V_0$ (for the case of a given speed on the press plunger).

Initial conditions:

$$\rho(q, 0) = \rho_0(q);$$

$$a(q, 0) = a_0(q).$$

The equations were reduced to dimensionless form and solved numerically using a conservative balance scheme. This scheme ensures exact (without taking into account the rounding error) fulfillment of conservation laws on any grid in a finite region containing an arbitrary number of nodes of the difference grid [10]. The grid is non-uniform in space and time. Then the resulting algebraic equations are solved using the sweep method.

As a result of the solution, unknown stresses ($\sigma_{zz}, \sigma_{rr}, \sigma_{\theta\theta}$), relative density ($\rho$), velocity ($V$), structural parameter (a) were found, which are functions of the coordinate (q) and time (t).

The problem has two scales of characteristic times: compaction $t_c = 4\mu_1/3P$, structuring $t_a = 1/k_{10}$, where μ_1 is viscosity of the incompressible base material, q_0 is the relative initial mass of the material, P is the pressure on the piston. Depending on the relationships between these scales, various modes of compaction and structural changes are possible.

Parameters (technological parameters and properties of polytetrafluoroethylene) vary within the following limits: $P = 10^7$–$5 \cdot 10^8$ Pa, $V = 8 \cdot 10^{-6}$–$2 \cdot 10^{-3}$ m·s^{-1}, $\rho_1 = 2.25 \cdot 10^3$ kg·m^{-3}, $\mu_1 = 10^7$–$4 \cdot 10^9$ Pa·s, $q_0 = 1.5 \cdot 10^{-2}$–$3 \cdot 10^{-2}$ m, $k_{10} = 1 \cdot 10$–$1 \cdot 10^{-3}$. The characteristic times corresponding to these parameter values vary in the following intervals: $t_c = 3 \cdot 10^{-2}$–$5.3 \cdot 10^2$ s, $t_a = 10^{-1}$–10^3 s.

3. Results and Discussion

3.1. Pressing mode with constant force on the press plunger

In general, the deformation mode is determined by the ratio of characteristic times. The limiting values of density and degree of structural changes are affected by the main parameters of the process.

Below are the results of numerical calculations for plunger cold pressing with a given force on the press plunger. Fig. 1 shows the dependences of density (ρ and structural parameter a on time t) for two limiting cases: the time of structural changes is much longer than the deformation time (Fig. 1*a*) and the time of structural changes is shorter than the deformation time (Fig. 1*b*). In the first case, the samples are dense, but not completely structured, and in the second case, the samples are not compacted to the maximum value.

Transitional modes of structuring and compaction lie between these extreme cases (Fig. 2).

It can be assumed that in the case when the characteristic time of complete structuring t_a is comparable with the deformation time t_c, the samples are smoother, but not compacted.

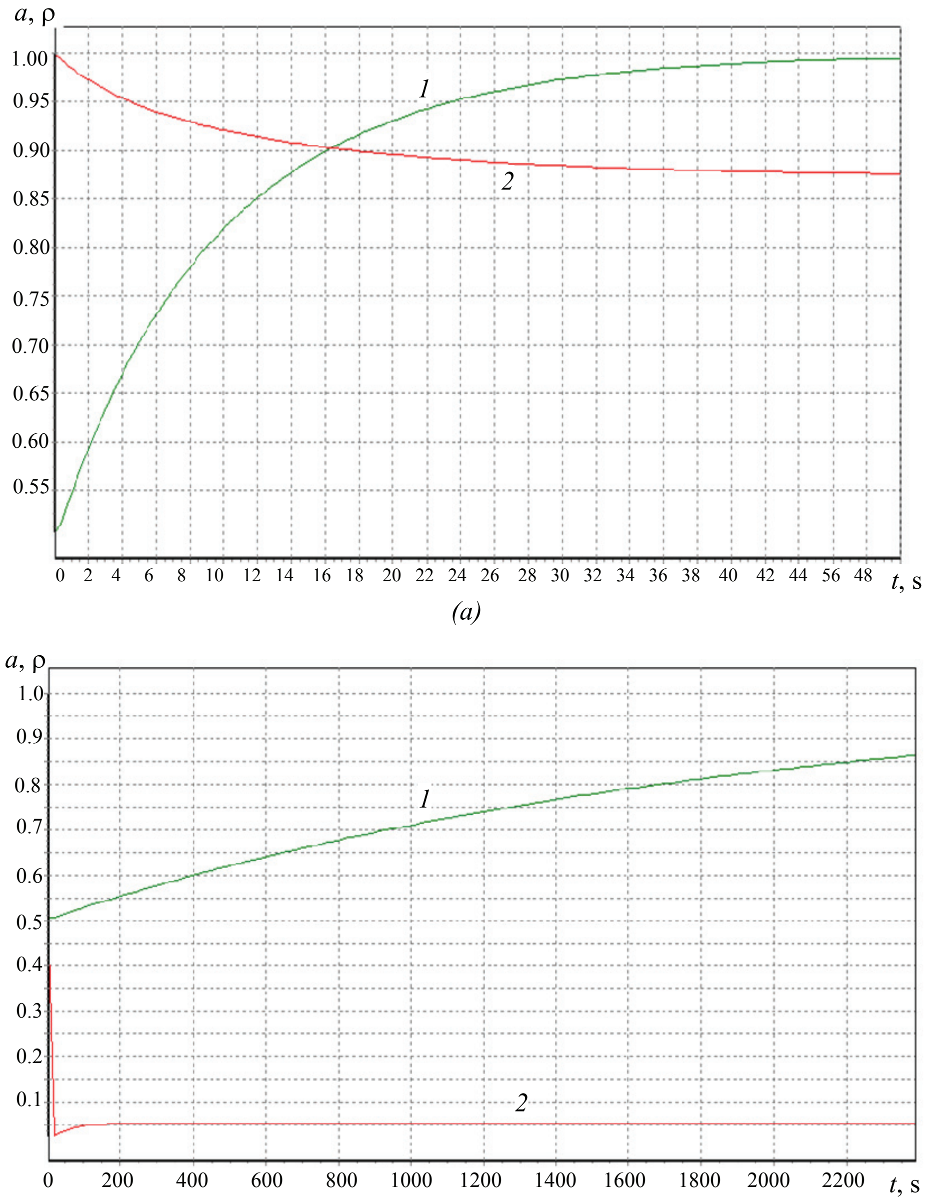


Fig. 1. Dependence of relative density ρ (curve *1*) and structural parameter *a* (curve *2*) on pressing process time *t*: $a - t_c << t_a$, $b - t_c >> t_a$

For the pressing mode with a given force on the press plunger, the dependence of the time to reach the maximum density value on the pressure on the press plunger is shown in Fig. 3*a*. It is evident that for pressure values less than 100 MPa, this time is very long (more than 25 min, and the characteristic compaction time is much longer than the structuring time. The material will be uncompacted.

The characteristic times of compaction and structuring strongly depend on the applied pressure on the press plunger (Fig. 3*b*). If the pressure is less than 100 MPa, the characteristic time of compaction is significantly longer than the structuring time, and when the pressure is greater than 130 MPa, the characteristic times are comparable. In these cases, the material is undercompacted and understructured. Between these modes (pressure from 100 to 130 MPa), the characteristic time of compaction is shorter than the structuring time, but the material has time to undergo these processes and the material should be of better quality.

3.2. Pressing mode with constant speed on the press plunger

When pressing in the mode with a constant speed on the press plunger, the material always reaches the ultimate density value [6].

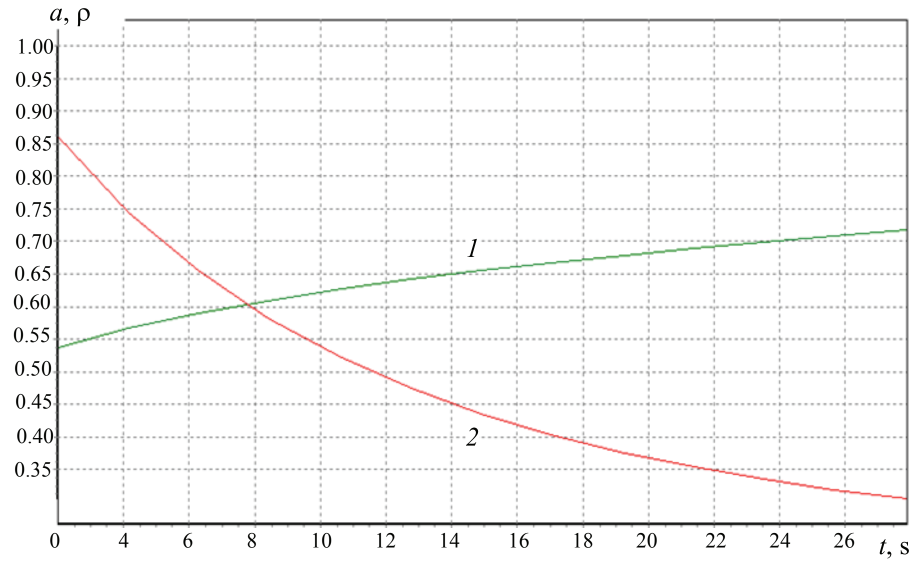
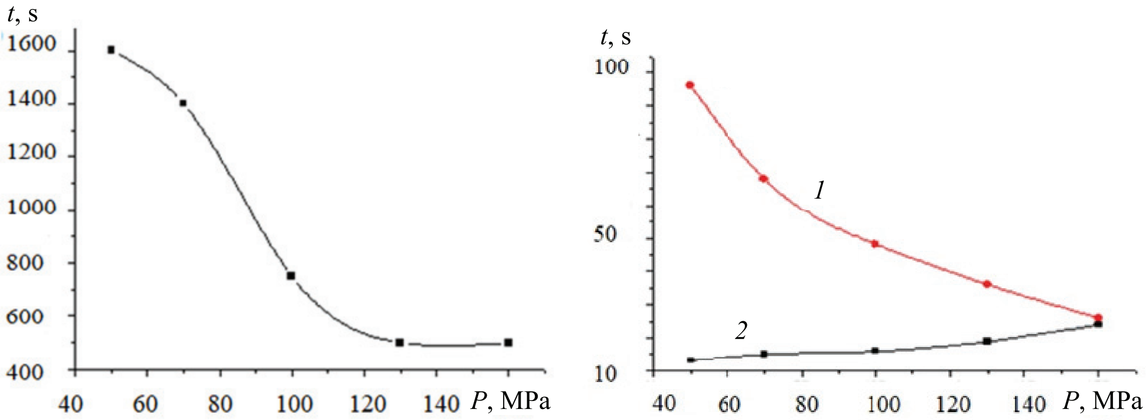


Fig. 2. Relative density ρ (curve *1*) and structural parameter *a* (curve *2*) versus pressing process time *t*: $t_c \cong t_a$



(a) *(b)*

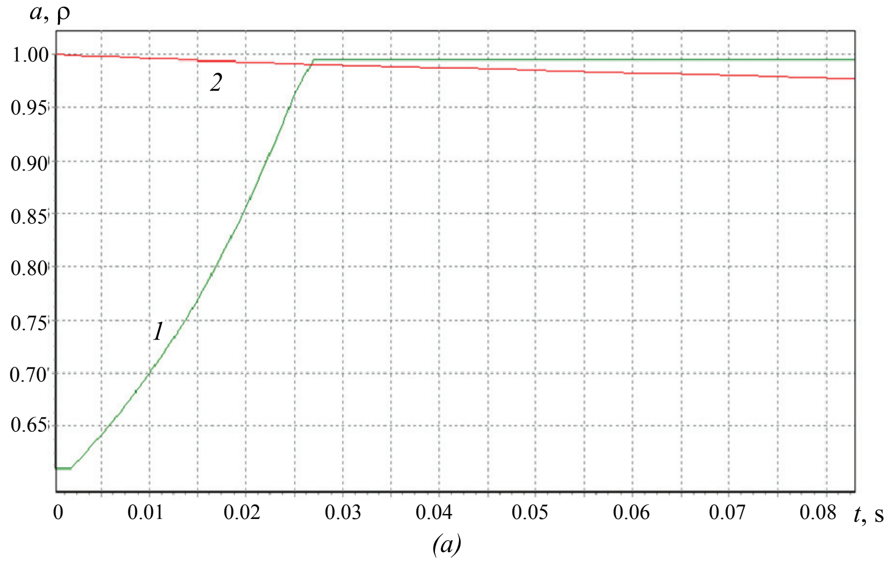
Fig. 3. Pressure dependence *P* on the press plunger:
a – time *t* to reach the limit value of density; *b* – characteristic compaction times (curve *1*) and structuring (curve *2*)

But in the case when the time of structural transformations is significantly longer than the compaction time, the material is compacted to the ultimate density, but the structuring process does not have time to complete (Fig. 4). In a large range of speeds (Fig. 3*a*, $V = 2\cdot10^{-3}$ m·s^{-1}, Fig. 3*b*, $V = 2\cdot10^{-5}$ m·s^{-1}) used in practice, this is exactly the case. In Fig. 4*a*, the characteristic compaction time (0.025 s) is much less than the structuring time (10 s), and in Fig. 3*b*, the characteristic compaction time (2.5 s) is slightly less than the structuring time (10 s), and in both cases, the density reaches its ultimate value, and structuring is not complete.

Figure 5 shows the case of pressing at a constant speed, when the characteristic times of structuring and compaction are comparable. It can be assumed that in this case the material will be less durable than in the case when the material is quickly compacted (Fig. 4*a*), although the structuring process is not finished.

The most acceptable case is when the compaction time is several times (3 or 4 times) longer than the structuring time. In this case (Fig. 4*b*) the material reaches the limit value, the structuring process is almost finished and the compaction process itself is smoother than in the case of high speeds on the press plunger, which should affect the quality of the obtained samples towards its improvement.

Figures 6, 7 show the dependences of the density and structural parameter on time for cases when the characteristic compaction time is slightly less than the structuring time ($t_c < t_a$) (Fig. 6), the characteristic compaction time is much less than the characteristic structuring time ($t_c << t_a$) (Fig. 7).



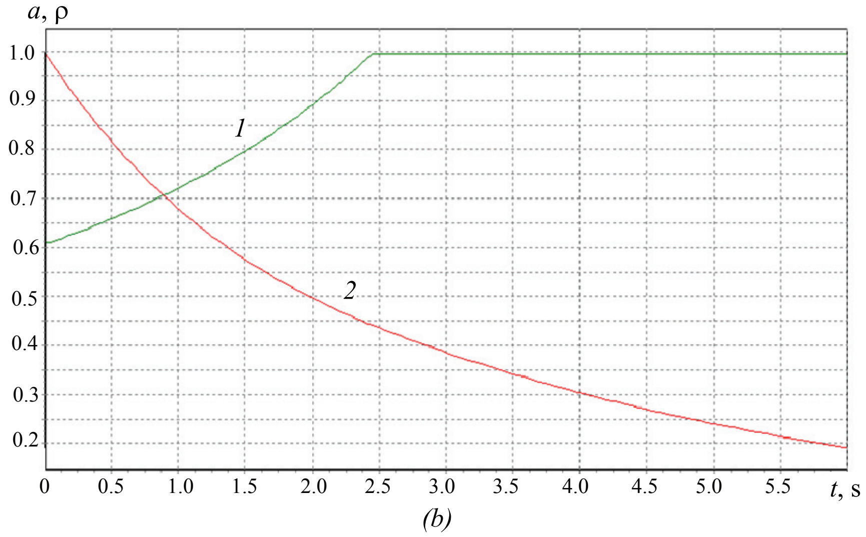


Fig. 4. Dependence of relative density ρ (curve *1*) and structural parameter *a* (curve *2*) on pressing process time *t*: a – $V = 2{\cdot}10^{-3}$ m·s^{-1}, $t_c \ll t_a$; b – $V = 2{\cdot}10^{-5}$ m·s^{-1}, $t_c < t_a$

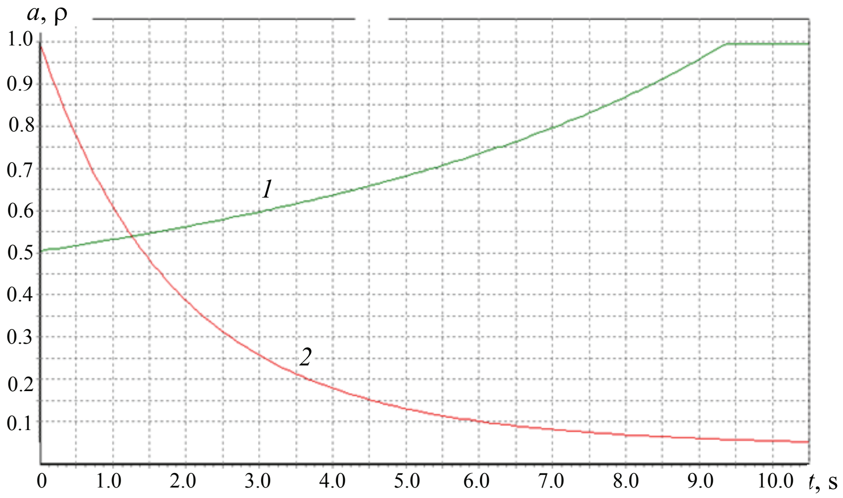
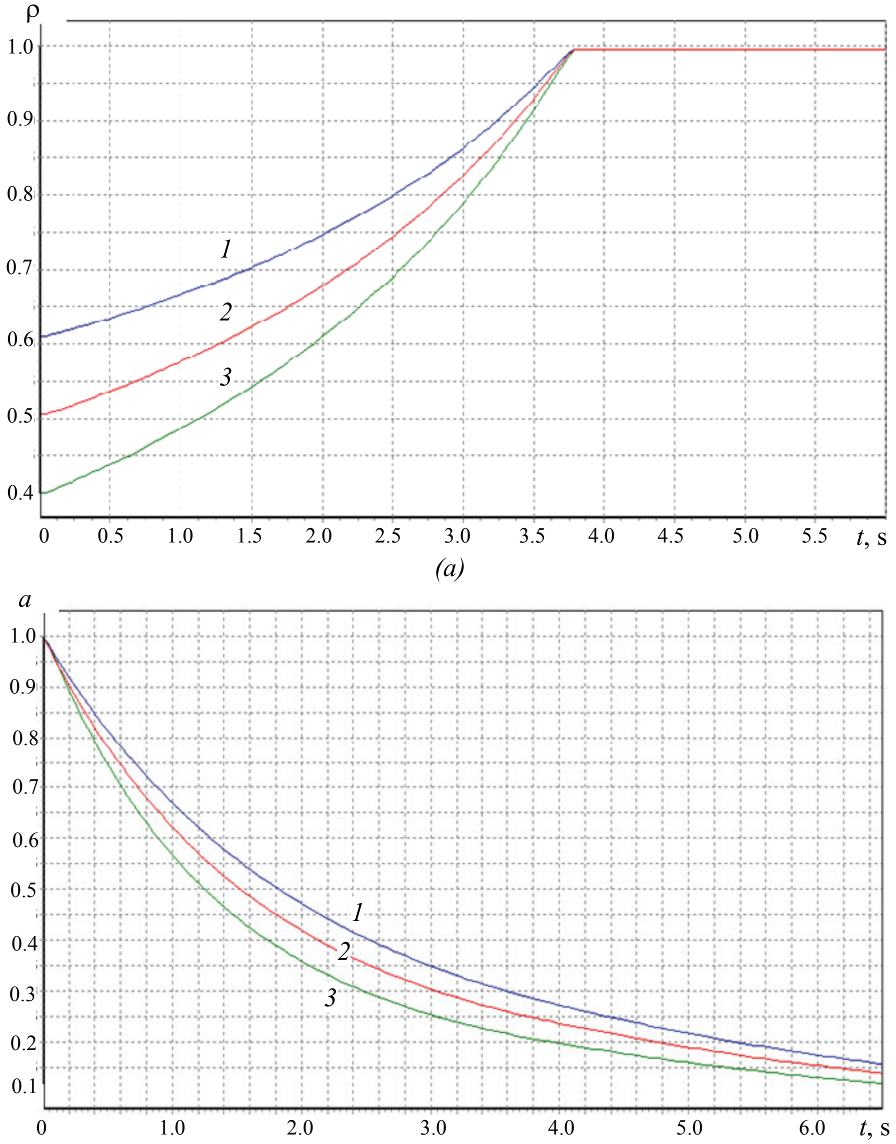


Fig. 5. Relative density ρ (curve *1*) and structural parameter *a* (curve *2*) versus pressing process time *t*: $V = 8{\cdot}10^{-6}$ m·s^{-1}, $t_c \cong t_a$



(a)

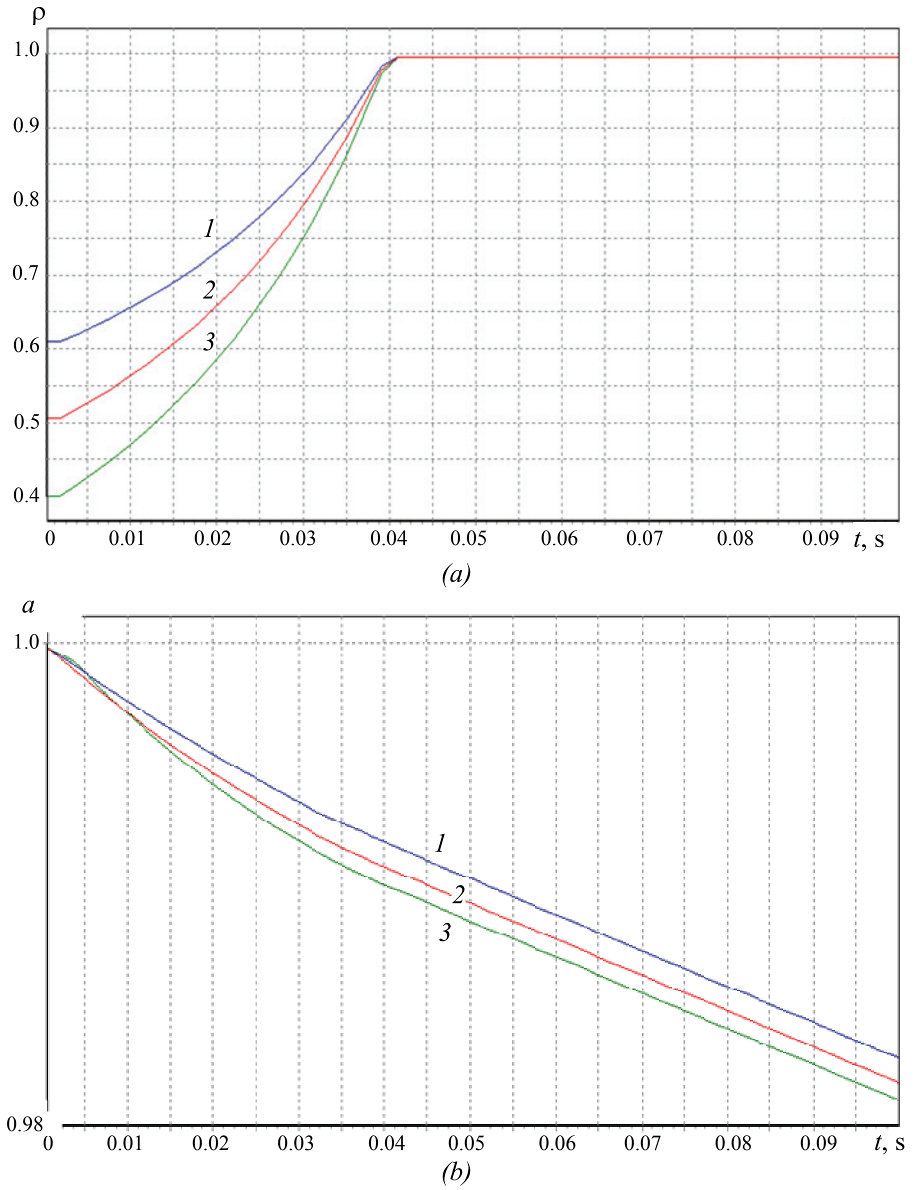
(b)

Fig. 6. Dependence of relative density ρ (*a*) and structural parameter *a* (*b*) on pressing process time *t* at three different points: top is a blue curve *1*, middle is a red curve *2*, bottom is a green curve *3*, $t_c < t_a$

In the first case, the material reaches the limit value of density and the end of structuring, and the difference in the value of the structural parameter of the top and bottom of the material by the end of pressing is no more than 0.05 (Fig. 7*a*). And for the second case, the structuring is not finished, and it can be assumed that there will be a non-uniform surface (the plunger speed in this case is $2{\cdot}10^{-3}$ $\text{m}{\cdot}\text{s}^{-1}$ = = 120 $\text{mm}{\cdot}\text{min}^{-1}$). Therefore, the option when the speed is 12 $\text{mm}{\cdot}\text{min}^{-1}$ is preferable (Fig. 6).

4. Conclusion

The development of the cold solid-phase pressing process depends on the ratio of the characteristic times of compaction and structural transformations. These times depend on the process parameters: the press plunger speed, the pressure on the plunger, the intrinsic properties of the material, the bulk and shear viscosities and their dependence on the density, the structural parameter, the geometry of the setup and the sample [11–14]. The averaging method used in this work and the application of Lagrangian coordinates make it possible to simplify the finding of the optimal region of parameters for the implementation of the most favorable modes from the technological point of view. For pressing materials in modes with a constant force on the press plunger and at a constant speed, such parameters should be selected when the time of structural transformations is slightly longer than the compaction time.



(a)

(b)

Fig. 7. Dependence of relative density ρ (*a*) and structural parameter *a* (*b*) on pressing process time *t* at three different points: top is a blue curve *1*, middle is a red curve *2*, bottom is a green curve *3*, $t_c \ll t_a$

Then the samples are dense, although the structuring process is almost complete, the distribution of the structural parameter and density along the height of the sample is uniform.

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6. Conflict of interest

The authors declare no conflict of interest.

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