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SHRINKAGE AND STRENGTH OF CAPILLARY-POROUS COLLOIDAL MATERIALS

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The processes of shrinking of capillary-porous colloidal bodies under the action of capillary-osmotic pressures have been investigated. The mechanisms of densification and strengthening of a material in the drying process have been considered. An equation relating the material's strength to its density, which agrees with experiment, is proposed.

Introduction. In the process of heat-and-mass exchange of capillary-porous colloidal bodies with the environment, changes in their volume and structure are observed. As the moisture is removed, the body makes a transition from the viscous-flow, plastic state to the solid state. This process causes a change in the internal porosity, creates unrelieved stresses inside the structure of the material, and strongly influences the intensity of the heat and moisture transfer in it. In this connection, the problem of investigating the process of structure formation in material drying to find optimal technological regimes of obtaining high-quality final products arises.

Shrinkage Mechanism. Capillary-porous colloidal bodies differ in composition and structure [1]. In the drying process their shrinkage occurs, which is especially marked in organic hydrophylic materials, most of which consist of high-molecular compounds (HMC) of different chemical compositions.

Many capillary-porous colloidal bodies are composites. Aside from the polymeric skeleton (matrix), they contain various kinds of fillers, including mineral particles.

In the drying process, a moist material shrinks and is densified under the action of the capillary and osmotic pressures. Capillary menisci are formed between hard particles capable of withstanding the Laplace pressure. The capillary pressure is also carried by swollen particles and osmotic moisture inside of them. In the process of moisture evaporation from menisci, the capillary pressure, which is transferred to particles, rises and leads to their displacement and a compaction of the whole capillary-porous colloidal body. The osmotically absorbed water replenishes the capillary menisci through the moisture films covering their surface.

In organic jellies (agar-agar, gelatin, etc.), because of the absence of a rigid frame (matrix) no menisci are formed. Due to the osmotic pressure water flows into the spatial matrix until the elastic forces arising from the frame extension counterbalance this pressure. As the water evaporates, due to the elastic forces in the spatial net of hydrogen bonds between the water molecules and the functional polar groups of macromolecules, there occurs a matrix densification leading to a shrinkage of swollen organic jellies. Shrinkage in the drying process proceeds until a strong surface layer of interbound macromolecules — a "crust" — is formed.

Subsequent removal of moisture from a jelly leads to a thickening of the crust due to the mass transfer of macromolecules by the liquid flow to its inner surface. On completion of the jelly drying, a strong layer of interbound macromolecules appears and the rest of the internal volume is usually a void. In capillary-porous colloidal bodies, depending on their structure, both the capillary and the osmotic mechanism of shrinkage manifest themselves to a given extent.

For open systems, in which a heat-and-mass exchange with the environment occurs, the total rate of decrease in the entropy [2] is equal to

$$dS/d\tau = d_eS/d\tau + d_iS/d\tau\ , \tag{1}$$

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where $d_eS/d\tau$ is the entropy flow caused by the energy and substance exchange between the system and the environment; $d_iS/d\tau$ is the total entropy production due to the irreversible processes (heat conduction, substance diffusion) inside the system. In the process of material shrinking, there occurs a structure ordering followed by a decrease in the entropy production rate of an open system $dS/dt < 0$. This condition is fulfilled only in the case where the intensity of the entropy outflow $d_eS/d\tau$ as a consequence of the moisture evaporation into the environment is higher than the entropy production rate $d_iS/d\tau$ inside it.

The processes of drying and shrinking of materials proceed at low rates. Therefore, in any local volume of the material for each time τ a quasi-equilibrium state characterized by certain thermodynamic parameters can be taken. With time, in the presence of heat inflow and moisture removal from the material these parameters change and in the material's microvolumes a sequential change of quasi-equilibrium states takes place. With such an approach one can use the equation of classical thermodynamics for each local volume V:

$$TdS = dU + dA \ . \tag{2}$$

At a constant heat flow into the sample steady-state temperature fields appear much faster than moisture-content ones. Therefore, once a constant temperature T has been attained in a local volume of the material $dU = 0$ can be taken and, consequently, $p \approx$ const:

$$dA = pdV = TdS \ . \tag{3}$$

If in the material at a constant pressure a change in the volume is observed, then relaxation of internal stresses occurs. In the absence of a deformation of the material nonrelaxing stresses arise in it. At the initial stage of drying of swollen organic materials, their intensive shrinkage and relaxation of stresses are observed. The latter is due to the micromolecular conformations and the particle shift into free spaces of the body. As the moisture is removed, the number of intermolecular contacts between the particles increases and the body being dried acquires mechanical strength. The shrinkage rate decreases, and in the material nonrelaxing stresses arise.

For the initial stage of drying, differenting Eq. (3) at $p \approx$ const, we obtain

$$p\,(dV/d\tau) = T\,(dS/d\tau) \ . \tag{4}$$

Since $dS/d\tau < 0$, we then have $dV/d\tau < 0$ — the shrinkage is followed by a decrease in the material volume.

In the process of sample dehydration, the moisture-loss intensity and, consequently, the shrinkage work increase from the center of the samples to their periphery. The decrease in the entropy in any local volume is due to the decrease in the mobility of bound moisture molecules in reference to their displacement in the bulk water as well as to the decrease in the mobility of hard particles as a consequence of the strengthening of intermolecular bonds between them or between the macromolecules and their associates as the moisture is removed (evaporates). Consequently, in the process of shrinking, as the entropy decreases, there is an increase in the ordering of the body particles, in the number of intermolecular bonds between them and, accordingly, in its strength. Therefore, the structure formation is represented as a process of sequential creation of equilibrium structures in the presence of nonequilibrium processes of heat and mass transfer and physicochemical transformations proceeding in them.

Thus, in the process of shrinking, the volume and shape of the capillary-porous colloidal body change. If the shrinkage rate considerably decreases, then the work of the external and internal capillary-osmotic forces is expended in changing the distances between the elements of the material structure — atoms, molecules, and their complexes — leading to a decrease in the potential energy of the interaction and to a local or a complete disruption of interparticle bonds.

The energy of interaction (Fig. 1) between atoms or ions of a solid body at temperature T_0 and in the absence of external load can be represented by different potential functions [3].

When the temperature is changed from T_0, to which there corresponds the initial potential function distribution with a maximum at $r = r_0$, to $T > T_0$, the atom receives energy $U_h = (T - T_0) = c\Delta T$. Because of the increase in the distance to r_1 (Fig. 1), as heat flows in, thermoelastic stresses appear in the material.

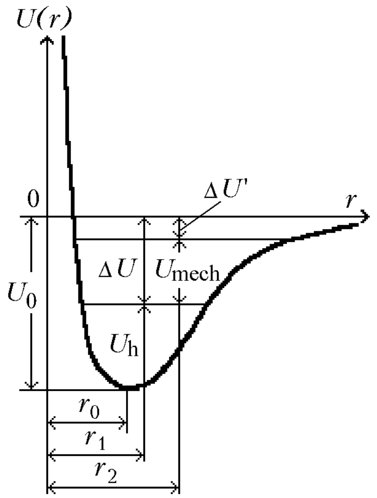

Fig. 1. Changes in the potential interaction energy of atoms $U(r)$ as a function of the distance r between them.

If a tensile force F is acting in a body (rod) of length l, cross section and relative deformation $d\varepsilon = dl/l$, then the specific work of the deformation $w = \int_0^{\tau} (F/s)\dot{\varepsilon} d\tau$. Within the limits of elastic deformations $\sigma = F/s = \varepsilon E$ and $w = E\varepsilon^2/2 = \sigma^2/(2E) = \varepsilon\sigma/2$.

At large stresses $\sigma = F/s$ or deformations ε, there occurs a disruption of interatomic bonds in the cross section having the maximum number of structure defects — continuity violations. The disruption is observed for a short time if large external loads are applied to the body. It can also take place at smaller loads but at large relative deformations ε and a large time of action τ of the external force. During this time ("longevity") there occur a repacking of the material particles, a redistribution of the external force over all interatomic bonds, their limiting lengthening, and subsequent disruption in the weakened cross section of the sample.

The value of w can be distributed over $n = \rho/m$ particles of mass m having one-dimensional tension. The work of the deformation per interatomic bond is equal to $U_{\text{mech}} = U/n = \varepsilon\sigma m/(2\rho)$. In the presence of U_{mech} there is a further increase in the interatomic distance to r_2 (Fig. 1).

At large mechanical loads F and temperature differences ΔT, the dependence $\varepsilon = (\sigma, T)$ is nonlinear. In the material, plastic deformations arise and they are accompanied by a relative displacement of the material particles and the formation inside the body of various kinds of defects.

The value of nonrelaxed stresses depends on the rate of increase in the external and internal pressures. The higher the rate of thermal treatment, drying, and shrinkage of the material, the larger the value of nonrelaxed stresses, which are different throughout the volume of the body.

A system containing nonrelaxed stresses is nonequilibrium. It tends, during a certain relaxation time, to the equilibrium state if the capillary-osmotic pressure considerably decreases (at low moisture contents — down to zero). This leads to an increase in the volume of the body, breaks (cracks) between individual blocks of a product in planes with the least number of cohesive bonds, and even to a break into small pieces (crumbliness).

Physicomathematical Model of Strength. The application of an external load and temperature changes within the limits of elasticity cause a linear relative deformation $\varepsilon = \sigma/E + \alpha\Delta T$. The values of E and α are assumed to be constant [4]. For a perfect (defect-free) cubic crystal lattice the one-dimensional relative deformation $\varepsilon = (r - r_0)/r_0$, where $r - r_0$ is the increase in the mean linear interatomic distance in the presence of nonrelaxed mechanical σ and thermal stresses. From this equality it follows that $r_0/r = 1 + \varepsilon$.

When heat is supplied to a solid body, the lattice atoms receive energy of a different value. Deviations (fluctuations) of the energy and local temperature T_{loc} of individual particles from the mean temperature T arise. A particle can receive a higher temperature by taking up energy from its surrounding atoms.

If the distance r slightly exceeds r_0, then the probability of bond breakage due to the thermal fluctuations is small. However, with increasing distances (Fig. 1, $r \geq r_2$) due to the thermal and mechanical action on the solid body the value of the energy barriers ΔU, $\Delta U'$ can decrease down to zero. As the mean temperature of the material increases, the probability of bond breakage increases due to the increase in the number of "hot" particles.

Assuming that the bond-breakage rate $d\chi/d\tau$ is proportional to the number of unbroken bonds $\chi_m - \chi$ in a perfect lattice, we obtain the equation

$$d\chi/d\tau = (\chi_m - \chi)/\theta \,, \tag{5}$$

in which

$$\theta = \theta_0 \exp (W/kT) \tag{6}$$

characterizes the mean time of bond breakage. The activation energy of bond breakage is equal to the potential function of the interatomic interaction at certain values of r or ε: $W = -U(r) = -U(\varepsilon)$.

Upon integration of Eq. (5) we have

$$\chi (\tau) = \chi_m [1 - \exp (- \tau/\theta)] \,. \tag{7}$$

Macrodestruction of a real body at a uniaxial deformation occurs during time τ in a certain cross section of the sample with the least number of cohesive bonds $\chi_r - \chi_m$.

From Eqs. (6) and (7) it follows that the longevity

$$\tau = \ln [\chi_m/(\chi_m - \chi_r)] \, \theta_0 \exp (W/kT) \,. \tag{8}$$

We assume T = const, $r > r_0$ (Fig. 1), $\varepsilon << 1$. For particles (atoms, ions) having a metal ($\nu \geq 1$) or ionic (ν = 1) bond [3], the function

$$W (r) = - U (r) \approx K\nu e^2/r = K\nu e^2/[r_0 (1 + \varepsilon)] \approx (K\nu^2/r_0) (1 - \varepsilon) = U_0' - \lambda\sigma \,,$$

where $U_0' = K\nu e^2/r_0$, $\lambda = U_0'/E$, $K = (4\pi\varepsilon_0)^{-1}$.

Substituting $W(r)$ into formula (8) and assuming $\tau_0 = \ln [\chi_m/(\chi_m - \chi_r)]\theta_0$, we obtain the Zhurkov equation $\tau = \tau_0 \exp [(U_0' - \lambda\sigma)/kT]$, which is in good agreement with the experiments performed with aluminum samples [5] and many other materials, in particular, oriented polymers [6, 7].

The links of high-molecular compounds interact with one another and other particles contacting them through the Van der Waals forces (Lennard-Jones potential) and covalent (Morse) bonds. Accordingly, these potentials can be represented at $\varepsilon << 1$ in the form

$$W (r) = - U_0 [(r/r_0)^{-12} - 2 (r/r_0)^{-6}] = - U_0 [(1 + \varepsilon)^{-12} - 2 (1 + \varepsilon)^{-6}] \approx U_0 (1 - 36\varepsilon^2) = U_0' - \kappa\sigma^2 = U_0' - \lambda\sigma \,,$$

$$U_0' = U_0 \,, \quad \kappa = 36U_0/E^2 \,, \quad \lambda = \kappa\sigma \,,$$

$$W (r) = U_0 \left\{1 - [1 - \exp [- a (r - r_0)]]^2\right\} = U_0 \left\{1 - [1 - \exp (- \gamma\varepsilon)]^2\right\} \approx U_0 [1 - (\gamma\varepsilon)^2] = U_0' - \lambda\sigma \,,$$

$$U_0' = U_0 \,, \quad \gamma = ar_0 \,, \quad \lambda = U_0\gamma^2\sigma/E^2 \,.$$

In the relations obtained, the values of U_0' and λ depend on the applied stresses σ, which is confirmed by experiment [7]. In the general case, at all values of ε, to calculate the activation energy W, it is necessary to make use of the atom-atomic potentials of the function $U(\varepsilon)$, which are specially selected to calculate each group of particular materials. Taking the logarithm of Eq. (8), we obtain dependences $\ln \tau = f(T^{-1})$ by whose angular coefficients the values of $W(r)$ are determined.

To calculate the hydrogen bonds, one can use the Morse and Lippincott potentials.

Real capillary-porous colloidal bodies consist of particles of different sizes, structures, and chemical compositions. These are moisture-saturated associates of macromolecules of different ordering, mineral particles of sand, clays, and other inorganic substances. Between particles and inside them, a wide spectrum of interatomic interactions arises. Usually the number of bonds having different interaction energies is larger inside the particles than between them. The strength of the body is determined by the number of cohesive bonds between contacting particles in its weakened cross section having, as mentioned above, the largest number of structural defects. Cohesive bonds arise between atoms in the surface layers of interacting particles.

If the body is isotropic in its strength properties, then such a material can be represented in the form of a quasi-cubic lattice made up of a large number of contacting polydisperse particles. It should be noted that the total interaction energy of all particles for isotropic bodies in the direction of the Cartesian coordinates will be equal.

The presence of the gas and liquid phases and inert fillers in a capillary-porous colloidal body decreases the number of cohesive contacts. As the moisture and air are removed in the process of drying and shrinking, their number increases and the strength increases.

Thus, the strength of various materials depends on the averaged energy of interactions between atoms of contacting particles in the direction of the action of the external load and on the number of contacts in the cross section with the largest number of continuity violations, i.e., dislocations, pores, cracks, and their changes under the action of the load. The above theory of destruction and strength of solid bodies can be applicable to such a model of a real composite material.

The strength of cylindrical samples of various materials is usually determined by breaking them with a press. The external load is transferred to the sample faces. Under the action of the axial load there is a decrease in the interatomic distances (compression) in the direction of the action of the force and their increase in the transverse direction (extension). With increasing distances bond breakage and displacements of the body particles in planes perpendicular to the sample axis are possible.

At compression the sample height decreases, but its cross section increases, reaching the maximum value in the middle of the sample. Such a barrel-shaped sample fails in the plane at an acute angle with its longitudinal axis. In this shear plane, the interparticle bonds of the material that are present in this cross section break. The body strength σ is proportional to the product of the probabilities of breakage of these binds in the direction of three axes of the Cartesian coordinate system $\chi_{rx}\chi_{ry}\chi_{rz}$, and for the isotropic body — χ_r^3.

We assume that at large deformations and loads the bond-breakage activation energy $W = U_0' - \lambda\sigma \to 0$ and in the cleavage plane $\chi_r << \chi_m$. On such assumptions the logarithmic function in Eq. (8) is ln $[\chi_m/(\chi_m - \chi_r)] \approx \chi_r/\chi_m$ and the number of disrupted bonds at a three-dimensional failure of the isotropic body is $\chi_r^3 \approx (\chi_m\tau/\theta_0)^3 [1 - 3(U_0' - \lambda\sigma)/kT]$. From this equality $\sigma \sim \chi_r^3$ follows. The value of χ_r depends on the number of contacting particles. Their number, in turn, is proportional to the dry substance mass in a unit volume of the wet capillary-porous colloidal body $\gamma_0 = \gamma/(1 + u)$. Consequently, $\chi_r \sim \gamma_0$ and $\sigma \sim \gamma_0^3$ or $\sigma^{1/3} \sim \gamma_0$.

Results and Discussion. As the object of investigation, we chose a characteristic capillary-porous colloidal body — mechanically dispersed peat.

Peat is a multicomponent system. It consists of an aggregate of organic and inorganic substances. The organic component of peat is a mixture of various high-molecular compounds — cellulose, hemicellulose, lignin, humic substances, bitumens, waxes, etc. The mineral component is represented by inorganic components of different dispersion distributed inside the inorganic mass. Mineral components make up a few percent of the total mass of dry peat.

Macromolecules of organic components of peat form associates. They contain sorbed and osmotic moisture. At contact points the associates interact with one another through cohesive (intermolecular) bonds. Between the associates there are spaces containing capillary water and "constrained" air.

The analytical dependence $\sigma^{1/3} \sim \gamma_0$ has been confirmed by a large number of experiments with cylindrical samples (pieces) of peat of different types, kinds, degrees of decomposition, specific surfaces, acidity, and with additives of surface-active substances [8, 9]. The present paper analyzes the results of the additional experiments performed under laboratory and field conditions.

As noted in [8, 9], the final strength of peat products is independent of the initial diameter of the sample upon its formation (Fig. 2a). It depends only on the number of cohesive interparticle bonds of the materials.

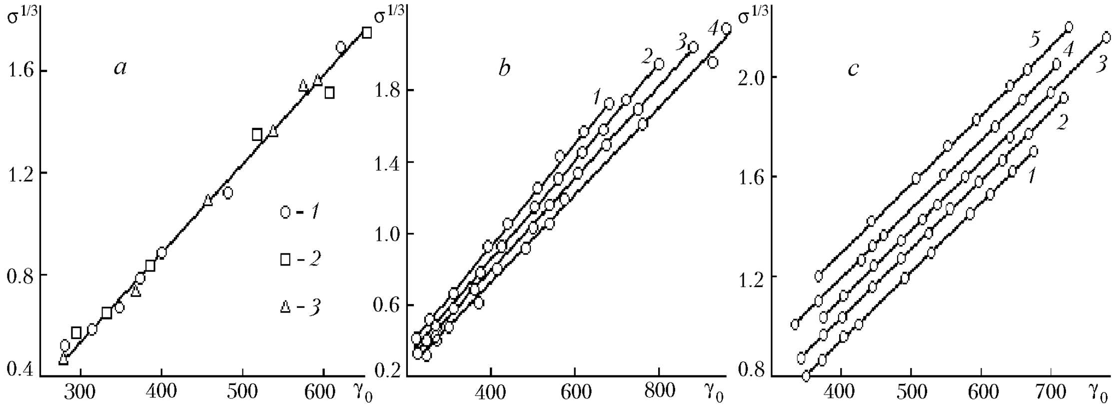

Fig. 2. Changes in the strength σ (MPa) of cylindrical samples as a function of the density of solid components in wet native peat γ_0 (kg/m^3); a) with initial diameter d = 20 (1), 30 (2), and 40 (3) mm and length 1.5d; b) with a specific surface of 380 (1), 470 (2), 510 (3), and 535 (4) m^2/kg; c) with initial moisture content u_{in} = 3.8 (1), 3.9 (2), 4.3 (3), 4.7 (4), and 4.9 (5) kg/kg.

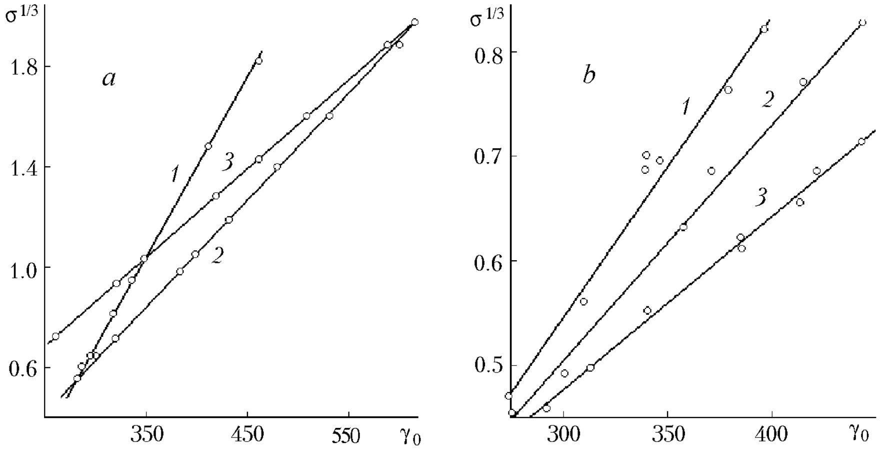

Fig. 3. Changes in the strength σ (MPa) as a function of density γ_0 (kg/m^3): a) of high-moor R = 10% (1) and valley R = 50% (2) peat and their 1:1 mixture (3); b) of valley sedge peat R = 20% (1) and R = 55% (2) and their 1:1 mixture (3).

Peat dispersion favors the obtaining of a stronger cylindrical piece. Dispersion of plant fibers leads to a breakage of the chemical bond between the links of macromolecules. The chemical bond is an order of magnitude larger than the hydrogen bond. Therefore, at a high degree of mechanical processing of peat there is a decrease in the mean energy of the bond between the microelements of its structure. Despite the increase in the number of cohesive contacts, a decrease in the total energy of intermolecular interactions leads to a decrease in the final strength of dried pieces of peat.

Linear graphs 1–4 (Fig. 2b) are similar but lie at different levels. This is due to the fact that the initial samples had different values of the specific surface, an initial density of the peat skeleton $\gamma_{0\text{in}}$ of respectively, 176, 191, 213, and 231 kg/m^3, a limiting moisture capacity of 9.54, 8.42, 7.8, and 77 kg/kg, and, consequently, a different number of cohesive bonds. In the drying process, the number of cohesive contacts increases. Because of the lower density and the smaller number of bonds, in the first sample the mobility of particles at shrinkage is higher than in the other samples. Therefore its strength increases more intensively. However, it is impossible to obtain thereby a denser structure in the process of shrinking and, accordingly, the final strength. In sample 4 at $\gamma_0 \approx 1000$ kg/m^3 the maximum final strength is observed.

The dependences $\sigma^{1/3}(\gamma_0)$ for pieces of peat at various values of the initial moisture content u_{in} are parallel graphs (Fig. 2c). Different initial values of the moisture content of the samples of one and the same peat were obtained by their preliminary drying. At lower values of u_{in} the samples had a higher initial density $\gamma_{0\text{in}}$. Therefore the strengthening mechanism is analogous to that considered in analyzing Fig. 2b. The final strength of a piece depends on the mobility of the particles forming it.

Experiments with peat pieces of initial diameter in the process of formation d = 10 cm and length 1.5d were performed under field conditions. The samples were obtained on a production extrusion-type moulding facility. With increasing degree of decomposition of peat equal strength is attained at a higher density of the material (Fig. 3a). An even higher density is obtained by mixing peat, of high and low densities (Fig. 3b). From these experiments it follows that the strongest pieces of peat are obtained from compositions made up of peat of different types, kinds, and degrees of decomposition. A definite combination of coarse fractions (plant remains — "reinforcement") and fine-grained ones (humus—"cement") favors the formation of stronger compositions that can be obtained by dispersing and mixing by special mechanisms. This permits obtaining a larger number of cohesive interparticle contacts of the material.

From the results of the laboratory and field experiments presented in this paper and in [8, 9] it follows that the angular coefficients η of the $\sigma^{1/3}(\gamma_0)$ graphs are practically independent of technological factors. This makes it possible to investigate the strength properties of pieces of peat depending only on the combination of its natural physicochemical factors. As a generalizing characteristic of the material strength, it is expedient to use the parameter η.

Conclusions. The mechanism of the elastoviscous-to-solid transition of wet materials in the process of their drying and shrinking has been investigated. The reasons for the development of relaxing and nonrelaxing mechanical and thermal stresses arising in the process of shrinking under the action of capillary-osmotic heat pressures have been considered. Breakage of the material occurs in a short time if a large external force is applied to it or slowly at a smaller force but a large deformation. A physicomathematical model of failure of solid bodies under the action of external mechanical loads and thermal fluctuations, which agrees with experiment, has been proposed. The material strength is determined by the number of cohesive contacts and the mean energy of the interparticle bond depending on the components of the capillary-porous colloidal body.

NOTATION

A, work, J; a, parameter entering into the potential Morse function, m^{-1}; c, atomic heat, J/K; E, Young modulus, Pa; e, electron charge, C; F, external force acting on the sample, N; k, Boltzmann constant, J/K; l, sample length, m; m, mass, kg; n, number of particles; p, total pressure equal to the sum of atmospheric, capillary-osmotic, and external mechanical (if any) pressures, Pa; R, degree of decomposition of peat, %; r, r_0, r_1, r_2, distance: any distance from the atom center corresponding to the potential function minimum (Fig. 1) at heating and tension, m; S, entropy, J/K; s, sample cross section, m^2; T, T_0, T_{loc}, current, initial, and local temperature, K; ΔT, temperature difference, K; U, internal energy, J; U_0, potential function minimum, J; U_{h} and U_{mech}, energy received by the atom due to the heat inflow and mechanical work (deformation), J; ΔU and $\Delta U'$, energy barriers, J; u, moisture content of the material, kg water/kg dry substance of the material; V, material volume, m^3; W, bond-breakage activation energy, J; w, specific work of the deformation, J/m^3; α, temperature expansion coefficient of the solid body, K^{-1}; χ, χ_{m}, χ_{r}, number of bonds: current value, maximum, and in the weakened cross section ("real"); ε, relative deformation; $\dot{\varepsilon}$, relative-deformation rate, sec^{-1}; ε_0, electric constant, Φ/m; γ, material density, kg/m^3; γ_0, mass of dry substance in a unit volume of wet material, kg/m^3; η, angular coefficient, Pa$^{1/3}$/(kg·m^3); λ, structural parameter; ν, ion valence; θ, relaxation time, sec; θ_0, period of thermal fluctuations of atoms, sec; ρ, solid-body density, kg/m^3; σ, stress, strength, Pa; τ, time, sec. Subscripts: loc, local; mech, mechanical; in, initial; r, real; h, heat; e and i denote the interaction with the environment and inside the body; m, maximum; x, y, z, Cartesian coordinate system.

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