

Dispersed-filled composites with a structured nanoscale

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Abstract. The article describes the new method for producing the asphalt concrete mixture, which has high strength characteristics, minimal water saturation. An attempt was made to theoretically substantiate the mechanism of hardening of the material. Asphalt concrete is a typical dispersed-filled composite material in which mineral particles of different dispersity are used as a filler, and the matrix is a binder-bitumen. To obtain a high-quality composite, it is necessary to ensure continuity of the material matrix. However, the formulations of asphalt-concrete mixtures used do not satisfy this requirement. This is due, on the one hand, to economic considerations, on the other hand, to a decrease in the strength of the material. The proposed method allows to solve this problem. The main consumer of bitumen in the asphalt mix is the smallest filler particles – mineral powder, which consumes up to 95 % of the binder. It is proposed to introduce a binder into the mineral powder by the granulation method by pelletizing. When the material moves in a rotating granulator, two types of forces act on the material particles: tearing off, caused by dynamic factors, and restraining forces caused by intermolecular interaction. Their combined effect makes it possible to form an ordered structure of the material with the finest interlayer layers of the binder between the solid particles realizing the nanoeffect. The structure thus obtained forms a continuous matrix of composite material with a minimum content of bitumen and strength indicators, several times higher than the requirements of GOST. The presence of a continuous matrix minimizes the water saturation of asphalt concrete, ensuring high durability of the road surface. The properties of the binder in the interlayers between the particles of the mineral powder differ significantly from the properties of the bulk bitumen, which prevents it from extruding at an elevated temperature from the structure of the material under the action of external transport loads, resulting in a consequent brittle failure. From the standpoint of the mechanics of a continuous medium, the stress state in liquid interlayers is represented by a set of spherical and deviator tensors, which made it possible to describe the mechanism of hardening of the material. The results of experimental studies are presented, which confirm the possibility of obtaining a qualitatively new asphalt-concrete mixture with characteristics that are substantially higher than the requirements of GOST, which is a worthy alternative to the traditional method of obtaining the material.



Keywords. Structured nanomatrix, disperse-filled material, asphalt concrete, asphalt concrete mixture, mineral powder, interphase layer, bulk phase

Introduction. In the process of operating a road, its road clothing and primarily asphalt concrete cover experiences increased loads from transport, temperature changes, weather conditions, ramming and other factors that lead to reduced durability, rapid loss of strength and the appearance of various types of deformation on the surface. At the same time, eliminating deformations in the process of repair work requires considerable financial costs. Today, special attention is paid to innovative road construction materials, modern technologies and methods of production [1], which allow to extend the service life, but, unfortunately, to create road clothes that would be absolutely crack-resistant impossible, due to aging and oxidation of bitumen, preparation of the mixture and production of works on its laying, instability of weather conditions, the presence of plastic deformations of asphalt-concrete coating, etc. It is necessary to strive for this.

Based on the considerable accumulated experience in the field of improving road building materials, and in particular in the field of asphalt-concrete improvement, a large number of works devoted to the modification of bitumens [2-6], the material that is the main linking element, can be distinguished. Material that determines the properties of the asphalt-concrete mixture and the finished asphalt-concrete coating. Hence, the improvement of asphalt-concrete mixtures is on the way to improving the indicators of bitumen. Unfortunately, there are not so many works devoted to the improvement of the technology of manufacturing the asphalt-concrete mixture [7] and this is due to the peculiarities of the production of the hot mixture. The preparation of a standard asphalt-concrete mixture consists of the following operations: preparation of mineral materials, preparation of bitumen, dosing of components, mixing of bitumen with mineral materials and unloading the resulting asphalt-concrete mixture into the body of a dump truck or a storage bin. During preparation, mineral materials are sorted by fractions, dried and heated to 200⁰C for subsequent mixing with mineral powder and bitumen. This technology of cooking has been practically unchanged for many decades and is not subject to serious modernization.

The purpose of this study is to describe the developed new technology for the production of asphalt-concrete mixture, which allows to obtain a qualitatively new material, with the subsequent theoretical justification of the results obtained in experimental studies.

The object of this study is asphalt concrete, which is a dispersed-filled nanocomposite in which road bitumen is used as a binding material, and mineral particles of different sizes and fractions, including crushed stone, sand and mineral powder serve as filler.

The subject of this study are processes and phenomena that describe surface phenomena from the standpoint of continuum mechanics.

The main objectives of the study are:

- analysis of the traditional technology of asphalt-concrete mixture preparation;
- development and optimization of the technology for the preparation of asphalt-concrete mixtures using a technological process – granulation by pelletizing;
- the formulation of theoretical provisions on the regularities of the formation of the structure of a dispersed-filled material on the example of asphalt concrete, which provides high shear stability and its strength characteristics;
- the theoretical estimation of the appearance of interphase forces in dispersed-filled nanocomposites on the basis of the known relations of the theory of elasticity.

Methods. Considering in detail the traditional asphalt-concrete mixture, it can be concluded that the main reason for the fragility of the pavement is the inability to distribute the binder evenly on the particles of the mineral part of the asphalt mix because of the limitations of the cooking technology itself. This is most pronounced for the smallest fractions of the mineral part, i.e. particles of a mineral powder. Mineral powder consisting of particles with a diameter less than a millimeter has a specific surface highly developed in comparison with other components of the mixture and requires up to 95 % of the bitumen injected into the material, according to the literature [8]. In accordance with the

requirements of GOST 9128-2013, the binder content in the material is 4-10 % of the mass of the mixture, and the mass of the mineral powder is 6-10 %, depending on the grade of the material. With this ratio of the components of the material, the principle of constructing dispersed-filled composites is violated, when the binder plays the role of a continuous structure matrix, and the filler particles form its dispersed part. At the indicated bitumen content, it is impossible to create a continuous matrix of the binder, which causes the presence of pores in the material, its increased water saturation and destruction during the off-season with alternating frosts and thaws.

Until recently, it was believed that mineral powder plays the role of passive filler space between large particles of material. However, P.V. Sakharov established the appointment of a mineral powder as a structural component, forming together with bitumen "asphalt-binding material", which adheres grains [8]. At a certain ratio of bitumen content and mineral powder in the system, a significant increase in bond strength between particles is possible, due to the structuring of the bitumen, i.e. translating it into a film state. At the same time, the strength indexes of structured bitumen significantly exceed the values of bulk bitumen, which leads to a significant increase in the strength characteristics of the material. According to P.V. Sakharov optimal concentration of bitumen in the binary system "bitumen - mineral powder" is 13 % by weight. In accordance with this, it becomes possible to create a high-strength composite material in which the matrix is an asphalt substance having higher strength characteristics than the binder. The main problem in solving this problem is the uniform distribution of small amounts of binder in the mass of the asphaltic substance. The degree of filling of space with monodisperse spherical particles is from 0.62 to 0.74 for various types of dense packages. Polydispersity of the material makes it possible to increase the value of this index due to the arrangement of fine particles between larger particles. Technologically this is possible with the use of highly intensive mixing devices, which, as a rule, leads to the appearance of air caverns in the mass of the processed material, which reduce the performance of the composite.

The problem under consideration was solved using a known technological method of granulation by pelletizing. The use of this technology for the production of asphalt-concrete mixture made it possible to obtain a fundamentally new material with high shear stability and strength. This method has been widely used on an industrial scale to produce granular mineral fertilizers. Granulation by pelletizing is carried out in rotary drum granulators. The granulated powdered material is introduced into the working volume of the apparatus and is wetted with a small amount of binder material. During the movement of the material along the walls of the drum, it is wetted and forms agglomerates of particles, onto which the powder grains roll to form round-shaped granules. The growth of the pellets is due to the alternate introduction of the powder fraction and the binder into the mass of the processed material.

Results. Application of the technology of asphalt concrete mixture preparation with the use of technological process – granulation by pelletizing made it possible to obtain asphalt concrete samples with high values of shear stability and strength. The composition of the asphalt-concrete mixture included mineral powder MP-1 with a weight content of 32.54 %, bitumen of BND grade 90/130 with a mass content of 4.96%, granite crushed stone cut of 2-5 mm fraction with a content of 62.5 %. The following indices were obtained: density – $2.49 \cdot 10^3 \text{ kg/m}^3$; compressive strength at 20°C – 4.3 MPa, which is almost double the requirements of GOST 9128-2013. For similar asphalt concrete, the strength should be not less than 2.5 MPa. The compressive strength at 50°C – 1.50 MPa. According to the requirements of GOST this indicator should be not less than 1.0 MPa; coefficient of water resistance – 1.02. According to the requirements of GOST this indicator should be not less than 0.95.

The formation of the ordered structure of the dispersed material in the granule is due to the nature of the forces acting on the particle during the formation and growth of the granule. Particles of the powder in contact with the surface of growing granules. In the course of such contact, two outcomes are possible: a) a particle of the powder adhered to the wetted surface of the granule; b) a particle of the powder came in contact with the granule, but was torn from its surface by the moving material. The realization of this or that outcome depends on the values of the powder acting on the grain. Here it is necessary to distinguish two types of forces: dynamic forces that seek to break a

particle from the wetted surface of the granule; forces that hold the particle on the surface. The ratio of these forces determines the outcome of the contact between the grain of the mineral powder and the granule.

Forces of the first kind are dynamic forces that arise as a result of the movement of material in the volume of the drum. The values of these forces depend on the mode of motion of the drum, its diameter, the level of drum loading with material, the properties of the material being processed, and many other factors. The holding forces are due to the action of the forces of intermolecular van der Waals attraction. The values of these forces depend on the properties of the wetting liquid and the nature of the interaction of the liquid and solid phases of the disperse system. The forces of intermolecular attraction vary within the thickness of the interphase layer and, with satisfactory wettability, reach a maximum value at the interface between the solid and liquid phases. With the distance from the boundary, the interaction weakens and at the contact boundary of the interphase layer with the bulk phase of the liquid becomes equal to the interaction of molecules in the bulk phase. In this region, the special properties of the liquid do not appear.

Thus, the value of the interfacial holding force will depend on the thickness of the wetting layer and the contact area of the powder particle with the granule. In this case, the smaller the thickness of the wetting layer, the stronger the grain of the powder will be attracted to the formed granule. The value of the attractive force will be equal to the product of the interfacial tension on the contact area of the powder particle and the granule. If there is insufficient contact area, the powder particle will be torn off the surface of the growing granule, and with good contact it will be included in its composition. This ensures an ordered structure of the granule material structure and a uniform distribution of the binder, eliminating the formation of air pores.

When using coarse-grained fractions of the mixture as embryos of granular material, it is possible to obtain an analog of a conventional asphalt-concrete mixture. The granulation process is carried out with the initial loading of large mineral particles into the granulator drum, and the granulation is carried out using bitumen and mineral powder. Such a method makes it possible to obtain an asphalt coating on each particle of the mixture in a guaranteed way. In the process of compacting the material, the plastic shells deform, filling the space between the coarse particles, forming a practically monolithic system. As studies have shown, to obtain such a system, 20 to 30 % of the mineral powder is required from the mass of the mineral part of the asphalt concrete. The technical solution is protected by a patent of the Russian Federation [9]. The structure of the material obtained by such a technology is a dispersion-filled composite in which the matrix is an asphalt substance.

Discussion. The effect of hardening of composite materials by introducing a fine-dispersed filler into the matrix has been known for a long time. The main provisions for the formation of ideal composite materials were formulated at the beginning of the last century by academician, the developer of the new scientific direction of physicochemical mechanics, P.A. Rebinder. He argued: "The easiest way to increase the strength of any solid to almost the ideal ceiling is to grind it to particles that are in order of magnitude corresponding to the distances between the dangerous weaknesses. If such particles are densely packed or glued with the finest, and therefore also high-strength after curing, interlayers, the resulting material will be dense, impermeable to liquids and gases, macro-homogeneous, high-strength and durable" [10].

According to the ideas of P.A. Rebinder on the surface of mineral particles, diffusional structured casings of bitumen are formed, the density and viscosity of which have the highest value directly at the bitumen-mineral interface. As the distance from this boundary, the viscosity and density of the bitumen decrease and in the transition zone of the structured shell into the free bitumen, the nominal values are assumed, i.e. those values that characterize the material under ordinary conditions (bulk bitumen). The difference between the properties of bitumen in thin layers and the properties of bulk bitumen is determined by the nature of the molecular interactions at the interface between the solid and liquid phases. Bearers of thin oriented layers of bitumen are particles of mineral powder, which has the most developed reacting surface.

Thus, the determining factor for increasing the strength of such composites is the thickness of the interlayer layers between the particles of the mineral powder. The value of this quantity can be determined from simple considerations. Knowing the percentage of binder in the asphaltic and the specific surface area of the mineral powder, an approximate estimate of the thickness of the bitumen interlayers can be obtained. According to the above data on the composition of asphalt, you can determine the volume of bitumen in a unit of volume of material, as well as the mass of mineral powder. The specific surface area of the mineral powder MP-1 according to the passport data is 250-450 m²/kg, which makes it possible to find its surface in a unit volume of asphalt material. The calculation made gives an estimate of the thickness of the interlayer layers between the powder particles of the order of 10⁻⁷ m.

This raises the question of the possibility of including such formations in the category of nanoobjects. By now, there are different points of view on what to attribute to nanoobjects. Thus, in the United Kingdom and the United States, the dimensions 10⁻⁷-10⁻¹⁰ m are assumed, in Germany it is less than 10⁻⁶ m. In some particular cases, the critical length scale for new properties and phenomena can be below 1 nanometer (for example, working with atoms) or be more than 100 nanometers (for example, reinforced polymer nanoparticles have a unique feature at about 200-300) [11]. This allows us to classify the interlayer layers of the binder in the asphalt material to the category of nanoobjects and explain the effect of a cardinal increase in the strength of the material.

If the mineral particles in the material are unordered, the strength of the bonds between them is determined by the strength of the matrix of the composite material, which at the previously indicated ratio of the asphalt concrete components does not always form a continuous phase. In the material under consideration, the matrix of the system consists of asphalt coatings on large filler particles, which when formed form an ordered structure on each particle of the mineral part. As a result, the strength of the bonds between the particles of the material increases and becomes higher than the strength of the binder material, which guarantees a general increase in the strength of the composite. The presence of nano-structures in the structure makes it possible to classify this material as a class of nanocomposites, and the main apparatus for studying such materials is nanomechanics, which has been developing intensively in recent decades. A detailed review of the methods of mechanics used to describe nanomaterials is given in [11].

The foregoing allows us to conclude that in order to clarify the mechanism of hardening of a material with a nanostructured matrix, it is sufficient to consider surface effects in the interlayer layers of the binder between the particles of the mineral powder. For this, it is necessary to pass to the level of interatomic or intermolecular interaction of the objects of the system under consideration. In the educational literature, these questions are considered in different courses [12], such as nonrelativistic quantum mechanics [13], quantum electrodynamics [14], quantum field theory [15], condensed state theory and electromagnetic fluctuations [16]. According to modern ideas, the forces of intermolecular interaction have an electromagnetic origin and are manifested in the form of exchange, multipole, fluctuation and some other interactions [17]. A consistent theory of intermolecular interactions could be constructed only on the basis of quantum-mechanical representations. Due to the quantum nature of the motion of electrons and nuclei, the solution of the problem of finding intermolecular interactions reduces, strictly speaking, to solving the Schrödinger equation for a system of interacting molecules. Such a problem can only be solved approximately [18].

Modeling the fluid in the area of its contact with a solid surface involves studying the features associated with the formation of an interphase layer in this region. The specificity of the interphase layer consists in the fact that a powerful intermolecular interaction appears in it, leading to the appearance of significant gradients of the parameters of the state of the medium. It is known that the properties of molecules in the surface layer differ substantially from those in the volume of the system, i.e. in the bulk phase. The molecules inside the bulk phase experience the same effects from all directions. As a result, the forces of attraction are mutually balanced and their resultant is zero. On the molecules that are in the interphase layer, forces of unequal magnitude operate from different sides [19]. The interphase layer borders on one side with the bulk phase, on the other - with a solid surface.

The values of the forces acting on the liquid molecule inside the interphase layer from the side of the boundary phases are usually different, which leads to the formation of a resultant of these forces directed perpendicular to the phase interface.

This leads to a distortion of the isotropic pressure field in the bulk liquid phase, which becomes anisotropic, and the pressure, which in the bulk phase is determined by the scalar quantity, becomes a stress tensor. A necessary condition for the existence of an interphase layer is the satisfaction of the conditions of its mechanical equilibrium, which implies the possibility of using the apparatus of continuum mechanics. In mechanics of continuous media, it is common to distinguish two classes of forces acting on the particles of the medium: three-dimensional (sometimes called mass ones) and surface ones [20]. By volumetric understand such forces that act on the elements of the volume, such as the forces of weight, electrostatic attraction or repulsion, the force of the electric or magnetic field. Surface forces include forces acting on surface elements, for example, pressure forces, internal friction (viscosity) forces in a medium, and the like. According to modern concepts, the forces of attraction are long-range and belong to the class of volume forces. The repulsive forces are considered as surface forces, and their long-range radius is assumed to be zero [20].

We note the fundamental difference between bulk and surface forces. The vector of the volume force is a single-valued function of the point of space and time, i.e. forms a vector field of force. Surface forces take at every point of space an infinite number of values depending on the orientation of the control surface, forming a tensor field. The individual components of the tensor depend on the choice of the directions of the coordinate axes, but the tensor represents an integral physical object expressing a certain state of the medium, and does not depend on the choice of the coordinate system [20]. The formation of a force field in the medium of the interphase layer is due to the action of both surface and bulk forces. Surface forces in this case are the pressure forces that satisfy the Pascal law for the liquid phase, the stress tensor, which contains three identical diagonal components. The field of volume forces is formed by forces of intermolecular attraction, the resultant of which, as indicated above, is different from zero.

We represent the stress tensor as the sum of two tensors – isotropic (spherical) and anisotropic (deviator) [20]:

$$A = \begin{bmatrix} \sigma_{xx} & 0 & 0 \\ 0 & \sigma_{yy} & 0 \\ 0 & 0 & \sigma_{zz} \end{bmatrix} = \begin{bmatrix} P_{ls} & 0 & 0 \\ 0 & P_{ls} & 0 \\ 0 & 0 & P_{ls} \end{bmatrix} + \begin{bmatrix} \sigma'_{xx} & 0 & 0 \\ 0 & \sigma'_{yy} & 0 \\ 0 & 0 & \sigma'_{zz} \end{bmatrix}. \quad (1)$$

In the written equation, P_{ls} denotes the pressure in the interphase layer caused by the action of bulk intermolecular forces. The index l corresponds to the liquid, s – to the solid phase. The components of the tensor A must satisfy the relations:

$$P_{ls} = \frac{\sigma_{xx} + \sigma_{yy} + \sigma_{zz}}{3}; \quad \sigma'_{xx} + \sigma'_{yy} + \sigma'_{zz} = 0. \quad (2)$$

The first term on the right-hand side of expression (1) defines the spherical part of the stress tensor, and the tensor, whose components are denoted by primes, is a stress deviator. The use of such a representation of the interphase stress tensor makes it possible to form a field of pressures that are variable in the thickness of the layer.

Consider a planar interphase layer and associate with it a Cartesian coordinate system in which the X and Y axes are arranged in the solid-liquid interface and the Z – axis is perpendicular to it. We write the expressions for the stress tensor components:

$$\sigma_{xx} = P_{ls} + \sigma'_{xx}; \quad \sigma_{yy} = P_{ls} + \sigma'_{yy}; \quad \sigma_{zz} = P_{ls} + \sigma'_{zz}.$$

The values of the components of the interphase stress tensor depend only on the coordinate z , and the axial symmetry of the problem allows us to write down the relations for the deviator components of the stress tensor:

$$\sigma'_{xx} = \sigma'_{yy}; \quad \sigma'_{zz} = -\frac{\sigma'_{xx}}{2}. \quad (3)$$

To determine the component σ'_{zz} , we use the boundary condition at the boundary of the interphase layer with the bulk phase of the liquid. At this point, the effect of molecular attraction forces on the side of the solid surface on the molecules of the interphase layer ends due to the limited range of these forces. The values of the parameters of the state of the medium here are equal to their values in the unperturbed liquid. It follows from this that all the components of the stress tensor, caused by the action of interphase molecular forces, vanish. This allows us to write down the expressions for the deviator components of the interphase stress tensor:

$$\sigma'_{zz} = -P; \quad \sigma'_{xx} = \sigma'_{yy} = \frac{P}{2}. \quad (4)$$

The result obtained allows us to establish the physical meaning of the surface tension of a fluid as a parameter, the value of which is determined by the deviator components of the stress-phase tensor. According to Bakker's formula [21], the surface tension γ is defined as the integral:

$$\gamma = \int_{-\frac{\delta}{2}}^{\frac{\delta}{2}} (P_N - P_T) dz,$$

where δ – is the thickness of the interphase layer, P_N – is the normal component of the pressure tensor, P_T – is the tangential component of the tensor, and z – is the coordinate along the normal to the surface of the interphase layer. In this case, the values of the components of the pressure tensor are not defined, and their physical meaning remains incomprehensible. As part of the study, surface tension can be defined as:

$$\gamma = \int_{-\frac{\delta}{2}}^{\frac{\delta}{2}} \sigma'_{xx} dz.$$

In the proposed approach, the mechanism of formation of intermolecular forces is not of decisive importance, and the fact that they exist is important. To develop a model of the interphase layer from the positions of the considered concepts, it is necessary to have the dependence of the molecular forces on the distance, and also the value of the radius of long-range interaction of these forces. In this situation, abstracting from rigorous theoretical calculations, it becomes possible to use the results of the experiment. Most of the best calculation methods use theoretical form equations with empirical correlations of constants that are not given by theory [22]. Thus, the van der Waals equation is a modification of the simple equation of state of an ideal gas $P_V = NRT$ [23]:

$$(PV + \frac{a}{V^2})(V - b) = RT, \quad (5)$$

where V – is the molar volume. The modification takes into account the fact that the pressure on the walls of the vessel P , produced by the molecules striking them, decreases due to the mutual attraction of the molecules. The constants a and b have some theoretical meaning, but it may be better to consider them empirical [23]. For van der Waals interaction, the region of distances from several angstroms to several thousand angstroms is characteristic. In this case, in the interaction of large macroscopic bodies, Van der Waals forces manifest themselves in a relatively narrow (although macroscopic scale) layer near the surface [12].

The distribution of pressure near a solid surface depends, first of all, on the ratio of adhesion and cohesion forces. With respect to the composite in question, it is assumed that the surface of the filler particles is well wetted by the liquid binder. This character of wetting indicates the excess of the adhesion forces over the cohesive forces. In this case, the force of molecular attraction will be directed toward the solid surface, forming an excessive pressure with respect to the bulk phase. The pressure distribution P over the thickness of the interphase layer δ is shown in Fig. 1. The dashed vertical lines indicate the boundaries of the interphase layers. Here we use the notation: P_s – pressure on a solid wall; P_l – the pressure in the bulk phase of the liquid; P_{ls} – the pressure at an arbitrary point in the interphase layer.

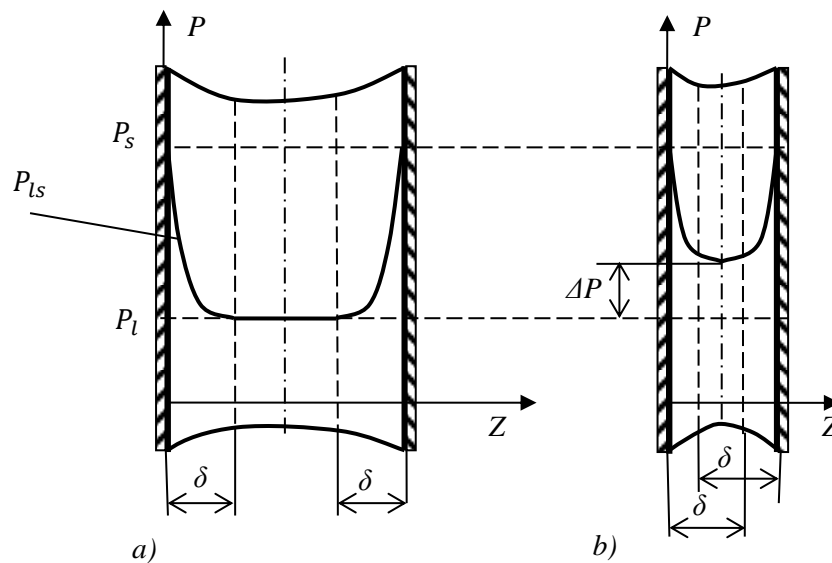


Fig.1. Pressure distribution in interphase layers:
a) without overlapping layers; b) when overlapping layers.

Figure 1(a) shows the distribution of pressure in a thick film when the interfacial layers are separated by a layer of the bulk phase. Figure 1(b) shows the case of overlapping of interphase layers, as a result of which the pressure in the middle of the interlayer increases by ΔP in comparison with the pressure in the bulk phase. Under pressure, one should understand here the internal pressure in a fluid caused by the action of the forces of intermolecular attraction. In the bulk phase, this pressure in accordance with the Van der Waals formula is equal to $P_l = a/V^2$. It is this pressure that determines the strength of the material of the liquid interlayer, and the increase in internal pressure in the binder means an increase in the strength of the composite as a whole.

Conclusions. Thus, the results of experimental and theoretical studies have confirmed the possibility of obtaining a high-strength nanocomposite material for paving on the basis of traditional components of the asphalt-concrete mixture with the use of a technological method – granulation by pelletizing. Asphalt agent in the form of pellets obtained by pelletizing allows obtaining asphalt concrete with a minimum water saturation, which ensures the durability of the road surface. Structuring of bitumen in the interlayers between the particles of the mineral part of the asphalt concrete prevents its extrusion at an elevated temperature from the structure of the material under the influence of external loads, leading to its brittle fracture. It should be noted that the proposed technology is applicable to the production of plastics with increased strength characteristics with a significant decrease in the content of polymers. In addition, the paper formulates approaches to describing surface phenomena from the viewpoint of continuum mechanics using the equation of state

of the medium in the form of the van der Waals equation. This approach does not pretend to be perfect, but it can be very useful for engineering calculations. More detailed information on the materials of the article can be found in publications [24] - [25].

Acknowledgements. We express our gratitude to all the colleagues who supported us while working on the article.

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