Influence of Carbon Primary Nano Materials in Polymer Modified Binders on the Temperature Sensitivity of Asphalt Concrete During Operation

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Abstract. The influence of the content of single-walled carbon nanotubes on the basic physical and mechanical properties of polymer-bitumen binders is studied. The effect of nanomodifiers on the morphostructural features of polymer-bitumen binders was studied using optical methods and fluorescence microscopy. The rational content of the modifying additive (0.0001 %) in the composition of the binder, which ensures the achievement of the specified parameters, is established. The influence of single-walled carbon nanotubes on morphostructural features is established. the nanomodifier initiates the processes of structuring interfacial layers, which leads to strengthening of physical bonds between the polymer macromolecules and the dispersed phase of the organic binder, which naturally affects the improvement of heat resistance, adhesive and cohesive strength of the binder. The study of polymer-bituminous binder films allowed us to record that when the nanomodifier is introduced, the polymer macromolecules decrease and their distribution over the binder volume is more uniform, which ensures its stability. It was found that the introduction of a rational amount of nanomodifier allows you to expand the required temperature range of asphalt concrete pavement performance by 25-34 %, while reducing the polymer content by 38 %.

1. Introduction
The state of the road network is an indicator that characterizes the quality of life of any state. Stable and long-term economic development depends on its quality, which determines the special attention of state departments to its condition. In modern Russia, roads with asphalt-concrete pavement prevail. High rates of motorization in the second half of the XX century led to the need to increase the pace of road construction and the availability of high-quality composite materials that can withstand the ever-increasing loads from moving vehicles. This predetermined the growth of various materials science studies aimed at studying and improving the properties of asphalt concrete and raw materials used for production [1-5].

The development of modern construction materials science is impossible without creating a new generation of materials that meet modern operational requirements. The most promising area of
materials science today is the control of structure parameters and properties of building composites by means of nanoscale modifiers: nanotubes and other carbon frame structures [2-9]. This can be achieved by using modern technological and prescription parameters of exposure, including at the molecular level, directly on composites or their component raw materials.

One of the most successful and widespread technologies developed to improve the performance of asphalt concrete is the modification of bitumen with polymer additives. These compositions are called polymer-bitumen binders (PBB). In general, it is bitumen modified with a polymer. The quality of the modification is determined by the ability of the polymer to improve the performance of bitumen when added in small quantities. In particular, the polymer modification is aimed at making the bituminous binder resistant to plastic deformations, increasing reversible deformations of the road surface at high temperatures, reducing temperature susceptibility, maintaining a sufficient level of workability during mixing in the production of asphalt concrete mixture and during laying, as well as providing lower levels of rigidity at medium and low temperatures in the road asphalt concrete surface. In addition to these parameters, polymer modified bitumen (PMB) must be stable during storage and resistant to aging [10]. However, such properties must be considered as the macroscopic result of complex thermodynamic and chemical interactions that occur between the bituminous binder and the polymer. In particular, the success of the modification depends on the possibility of polymer swelling in a bituminous environment. Polymer phase by means of absorption of light bitumen fractions, forming a high compatibility with the bitumen. And in the meantime, it retains its own structure. These conditions occur at an adequate degree of swelling and solubility of the polymer in the bituminous phase [11].

In the Russian Federation is spread to the application of bitumen obtained by oxidation, they are characterized by a low content of light tar fractions and a tendency to aging, which causes a number of difficulties in the development of high performance polymer modified binder received (MBP) displayed a number of shortcomings, such as peellability, due to the fact that it is a multicomponent system, and a tendency to ageing and low adhesion.

One of the methods for improving the properties of polymer-bitumen binders, which allows controlling the parameters of properties at the molecular level, is the use of nanostructured modifying additives (nanomodifiers) [1, 4, 7]. This method allows you to create a new generation of composites, devoid of the above disadvantages, with improved characteristics that can be used to obtain high-strength structural, insulating building materials. For modification of polymer composites, carbon primary nanomaterials are most often used-carbon nanotubes (CNTs) and fullerenes, which have unique physical and mechanical characteristics, for directed structure formation of building composites [2].

CNTs have characteristics that indicate the prospects of their use in the construction industry: colossal strength; high values of specific surface area and specific surface energy; inertia in relation to any acids and alkalis [4]. Today, many varieties of CNTs are known, but there are also some difficulties that make their use difficult: the lack of reliable information about the impact of nanoscale particles on the environment and human health; imperfection of technological and laboratory equipment; the skeptical attitude of society to nanotechnology, characterized by misunderstanding and distrust; the increased tendency of CNTS to aggregation, which makes it difficult for them to be evenly distributed over the entire volume of the composite, etc. The latter fact does not allow us to fully reveal the possibilities of CNTS in the composite, and use, for example, their high modulus of elasticity and strength at a very low density [12].

Currently, the most important tasks in obtaining stable nanostructured polymer composites are to ensure the aggregate and sedimentation-stable state in the "carrier-carbon primary nanomaterial" system and the uniform distribution of this system in the polymer matrix. To solve the first problem, it is necessary to destroy aggregates of primary carbon nanomaterials and ensure the neutralization of excess surface energy – solvation. Then, evenly distribute the "carrier-carbon primary nanomaterial" system in the matrix, which is usually complicated by the high viscosity of the polymer. The second task is to ensure the necessary surface interaction at the matrix-filler interface, otherwise the physical
and mechanical properties will not be sufficiently realized. The identified problems and their solutions formed the basis for the study.

2. Research materials and methods

The objects of research were: the source material of single-walled carbon nanotubes (SWNTs) obtained by thermal evaporation of graphite in the presence of Ni-Cr torus catalysis in an electric arc (Arc SWNTs), containing graphite nanoparticles 20-100 nm long and metal nanoparticles 5 nm in diameter, produced by the Russian Academy of Sciences Chernogolovka. Samples of polymer binders were prepared on the basis of BND 60/90 bitumen produced by Moskovsky Oil Refinery. The polymer used was divinyl-styrene-styrene-butadiene styrene SBS 30L-01, produced by Voronezhstintezkauchuk LLC.

The influence of the nanostructuring component and the polymer and the determination of optimal contents were determined by varying the concentration of the polymer and SWNT in the range of 2.8-4.0 % and 5·10⁻⁴...1·10⁻³%, respectively. The range of variation of the count is limited by the weight accuracy of the dosage of the analytical balance VLR 200. The content of the plasticizing medium—carrier for carbon primary nanomaterials was determined in advance when selecting the control composition of the polymer-bitumen binder that meets the requirements of state standard it was 2.5 % [13].

Preparation of PMB was carried out by three-stage technology, the first step was preparing the system "carrier-primary carbon nanomaterial", at the second stage was preparing nenormirovannym matrix polymer component; in the third stage, the polymer component was introduced to the bitumen and mixed until a homogeneous effective polymer-bitumen binder. A similar approach in the preparation of PBB was used to enable the assessment of the impact of SWNTs and the uniformity of their distribution in the polymer matrix.

The first stage: preparation of the "carrier-carbon primary nanomaterial" system was carried out by combining single-walled carbon nanotubes with a plasticizing medium with a carrier and subsequent dispersion using an ultrasonic dispersant of the Vibra Cell Sonic750 immersion type, figure 1. The content of SWNTs varied in the range of 5·10⁻³...1·10⁻⁴ % by weight of the finished polymer modified binder.

![Figure 1](image)

**Figure 1.** Figure with schematic representation of the preparation of the «carrier-carbon primary nanomaterial (SWNTs)" system».

Second stage: Preparation of the nanoarmed polymer matrix was carried out by introducing a polymer into the "carrier-carbon primary nanomaterial" system. When a polymer is combined with a nanomodified plasticizing medium, its swelling processes occur, with simultaneous adsorption of hydrocarbon compounds of the carrier medium. The third stage: combining and mixing the nanoarmed polymer matrix with a bituminous binder until a uniform distribution of the polymer in the volume is achieved. On the hydrocarbon chains of polymer macromolecules, a part of light
hydrocarbons from the maltenic medium of bitumen is adsorbed, thereby slowing down the transition of oils to resins and the formation of asphaltenes. The uniform distribution of polymer units contributes to achieving equilibrium in the solution and stability of the composite prepared on its basis.

The rational parameters of the preparation of the "carrier-carbon primary nanomaterial" system were determined using an integrated approach based on the determination of the optical density and refractive index using a KFK-3 Photometer and an IRF-22 Refractometer, respectively.

Optical density $D$ is a measure of the opacity of a substance layer for light rays. It is calculated as the decimal logarithm of the ratio of the radiation flux $F_0$ incident on the layer to the attenuated as a result of absorption and scattering, the flux $F$ passed through this layer:

$$D = \lg \left( \frac{F_0}{F} \right).$$

The refractive index is equal to the ratio of the speed of light in a vacuum to the speed of light in a substance, i.e. it shows how many times the speed of light in a vacuum exceeds the speed of light propagation in a substance.

The uniformity and homogeneity of the nanomodified polymer matrix was evaluated using an inverted industrial microscope Nikon Eclipse MA200, which provides the use of an integrated artificial intelligence system for automatically combining the obtained images with observation data using the light and dark field method. In our case, the light and dark field method was used.

3. Results and discussion
To assess the effect of ultrasound on the plasticizing medium, it was subjected to idle dispersion, that is, without a nanomodifier. There were no changes in the optical density and refractive index before and after exposure. To obtain more accurate results, samples were taken from three layers: upper, middle, and lower (figure 2). During the preliminary experiment, it was found that the concentration of CNT in the considered range of $5 \times 10^{-3}...1 \times 10^{-4}$% of the mass of the bituminous binder does not affect the technological parameters that ensure the uniformity of nanomodified plasticized media. The same dynamics of changes in optical density and refractive index is observed over the entire concentration
range. The obtained dependences of changes in the studied indicators are presented on the example of their content of $5 \cdot 10^{-4}\%$ for out (figure 3).

Figure 2. Sampling to determine the optical density and refractive index.

Figure 3. Effect of system dispersion time «carrier-carbon primary nanomaterial (SWNTs)» system on optical density and refractive index.

The results shown in figure 3 indicate that for two minutes of dispersion, the optical density and refractive index are equal in all three layers of the test sample, and in this time interval, the maximum value for the parameters under study is observed, which indicates that the minimum diameter of single-wall carbon nanotubes particles is reached and the logical increase in the specific surface of nanoparticles in the volume under study, which is reflected in the achievement of the maximum optical density value, as a measure of the opacity of the substance layer.

After determining the optimal dispersion time of the "carrier-carbon primary nanomaterial" system, a polymer (hereinafter referred to as the polymer matrix) was introduced into it, and mixing was performed until a homogeneous state was achieved. The polymer matrix was studied for uniformity and uniformity based on the results of microscopic survey of light and dark field film samples obtained by the method of light and dark field film samples shown in figure 4.

Figure 4. Microphotograph of the polymer matrix without SWNTs, multiplicity x 1000, using the light and dark field method.

Figure 5. Microphotograph of the polymer matrix with SWNTs, multiplicity x 1000, using the light and dark field method.

Figure 5 shows an inhomogeneous structure, indicating an uneven distribution of the polymer component in the plasticizing medium-carrier. When modifying the polymer matrix (Fig. 5), the process of nanostructuring of interfacial layers occurs, and the SWNT changes its structure. A more
uniform distribution of the polymer in the plasticizing medium becomes possible, as evidenced by the image, which shows insignificant objects made of undissolved polymer.

It is natural to assume that multi-factor mixing of SWNTs first in the plasticizing medium, then when combined with the polymer and at the final stage as part of the polymer matrix in the volume of bitumen, will contribute to a more uniform distribution of SWNTs in the volume of bitumen, and the visible structuring of the polymer matrix at the preliminary stage will give the PMB a set of useful properties. This was confirmed by laboratory work during an experiment to study the properties of modified PMB [11, 14, 16].

Control composition #1 was selected in the previous study [8]. Therefore, in this work, it was necessary to study the effect of SWNTs on the properties of BMP while reducing the polymer content from composition No. 1, due to the strong structuring ability of SWNTs at the stage of studying the polymer matrix. The compositions and properties of the studied samples of polymer modified binders are presented in table 1 and 2, respectively.

### Table 1. Compositions of the studied samples of PMB.

| The component name         | No 1   | No 2   | No 3   | No 4   | No 5   |
|----------------------------|--------|--------|--------|--------|--------|
| SBS 30L-01, %              | 4.5    | 2.8    | 2.8    | 2.8    | 2.8    |
| SWNTs, %                   | -      | -      | 0.005  | 0.0005 | 0.0001 |
| Plasticizing medium, %     | 2.5    | 2.5    | 2.5    | 2.5    | 2.5    |
| BND 60/90 bitumen, %       | other  |        |        |        |        |

### Table 2. Temperature range of PMB operability.

| The component name         | BND 60/90 | The requirement of state standard 52056-2003 | No 1   | No 2   | No 3   | No 4   | No 5   |
|----------------------------|-----------|-----------------------------------------------|--------|--------|--------|--------|--------|
| Softening temperature, °C  | 51        | no less 54                                    | 78     | 56     | 62     | 68     | 80     |
| Britteness temperature by Fraas, °C | -17 | no more -20                                  | -26    | -21    | -23    | -25    | -29    |

The obtained results were achieved thanks to the processes of structuring the polymer system at the molecular level. Based on the results of fluorescence microscopy of PMB films, the effect of the influence of SWNTs on changes in the structure parameters of polymer modified binders was determined, figure 5.
The presented results (figure 5a) show that the structure of the PMB sample (composition No 2) is composed of larger clusters of polymer macromolecules that are unevenly distributed over the volume. When the PBB (composition No. 5) is added to the OUN (Fig. 2B), there is a decrease in the polymer macromolecules and a more uniform distribution of them over the binder volume – structuring the system, which helps to ensure its stability. A structured composite matrix is formed, which provides high softening temperatures and brittleness of nanomodified PBB, due to the strengthening of structural bonds.

The obtained dependences of properties (table 2) should naturally affect the performance of the asphalt concrete pavement made using the developed PMB. The calculation of the required and required operating temperature range of asphalt concrete pavement, sufficient to ensure a given service life in specific natural conditions, was carried out using the method of Ya.N. Kovalev.

The estimated winter temperature of crack resistance is determined by the formula:

$$t_{0\text{min}} = 0.7 \cdot t_{b\text{min}}$$

where $t_{0\text{min}}$ is estimated winter temperature of crack resistance of asphalt concrete pavement, °C; $t_{b\text{min}}$ is absolute maximum winter outdoor temperature for the coldest days in Moscow OS, (table.3 BR 131.13330.2018), for Moscow this is 35°C.

Estimated winter temperature of crack resistance: $t_{0\text{min}} = 0.7 \cdot (-35) = -25$°C.

The calculated summer temperature of the shift resistance of asphalt concrete pavement using polymer-bitumen binder is the temperature of the road surface in July, which is determined in accordance with BCR 23.0-99, according to the formula of Ya.N. Kovalev:

$$t_{b\text{max}} = J \cdot (1 - A) \frac{K}{\alpha_c} \cdot t_{abc},$$

where $J$ is estimated total solar radiation flux for the operating area, kcal/(m²h) (paragraph 8 BR 131.13330.2018), for Moscow this is 418 kW·h/m² = 481 kcal/(m²h); $A$ is albedo coating (reflection coefficient), by Ya.N. Kovalev this is 0.11 for asphalt concrete; $\alpha_c$ is total calculated heat transfer coefficient of asphalt concrete pavement, kcal/(m²°C);

$$\alpha_c = \alpha_u \cdot \alpha_k,$$

where $\alpha_u$ is heat transfer coefficient due to radiation; for asphalt concrete surfaces according to V. M. Sidenko $\alpha_u = 3$ kcal/(m²°C); $\alpha_k$ is heat transfer coefficient due to convection, kcal/(m²°C):

$$\alpha_k = 4.23 \cdot V^{0.66} + 3.2 \cdot e^{1.28V},$$

where $V$ is wind speed over the surface for the operating area, m/s, (5 m/s for II RCZ) $\alpha_k = 4.23 \cdot 5^{0.66} + 3.2 \cdot e^{1.28 \cdot 5} = 12.24$ kcal/(m²°C).

Thus, the total calculated coefficient will be equal to: $\alpha_c = 3 + 12.24 = 15.24$

Coefficient that considers the part of the heat absorbed by the coating that is given to the surrounding air:

$$K = 0.15 + 0.052V,$$
$K=0.15+0.052V=0.15+0.052\cdot5=0.41$.

Air temperature in the boundary layer of the coating $t_{abc}$, °C, when the wind speed is from 0 to 1.0 m/s, it is determined by the formula obtained from the graphic data of Ya. N. Kovalev:

$$t_{abc} = (t_{b\max} + 20) - 12 \cdot V,$$

where $t_{b\max}$ is absolute maximum outdoor temperature in July, °C, (table 4, BR 131.13330.2018), for Moscow this is $+38^\circ$C.

Air temperature in the boundary layer of the coating: $t_{abc} = (38+20)-12 \cdot 1= 46^\circ$C.

Estimated summer temperature of asphalt concrete pavement shear resistance: $t_{\max} = 481(1-0.11) \cdot 0.41/15,24+46 = 57.5^\circ$C.

According to the method used, for Moscow, the temperatures of crack resistance and heat-shear resistance of asphalt concrete pavement are $-25^\circ$C and $+57.5^\circ$C respectively. From table. 2 it can be seen that the developed nanostructured composition of PMB (sample No. 5) exceeds the necessary requirements for the temperature range of the composite performance: by 14 % for the brittleness temperature and by 30 % for the softening temperature. The obtained data characterize it as a composite with increased resistance to plastic deformations in the summer and cracking in the winter. This was also confirmed when studying the properties of g-type asphalt concrete samples based on three binder samples (compositions No 1, No 2, No 5). The choice of the type of asphalt concrete is justified by the fact that composites that do not have a strong frame made of a large mineral skeleton are characterized by a high sensitivity of the binder in its composition to the perception of loads and temperatures. This will allow us to evaluate the contribution of the modified binder in the properties of asphalt concrete prepared on its basis. The results of the comparative experiment are presented in table 3.

**Table 3. Physical and mechanical characteristics of asphalt concrete type G, grade II.**

| Name of the indicator                  | The requirement of state standard 9128-2013 | No 1 | No 2 | No 3 |
|---------------------------------------|---------------------------------------------|------|------|------|
| Water saturation, %                   | 1.5…4.0                                     | 1.80 | 2.30 | 1.80 |
| Compressive strength, MPa at a temperature of: | | | | |
| +20°C                                  | >2.20                                       | 4.60 | 4.20 | 4.90 |
| +50°C                                  | >1.20                                       | 2.30 | 1.70 | 2.60 |
| +60°C                                  | -                                           | 1.60 | 1.10 | 2.10 |
| 0°C                                    | <12                                         | 8.60 | 10.00| 7.20 |
| Water resistance                       | >0.85                                       | 0.98 | 0.90 | 1.00 |
| Water resistance under long-term water saturation | >0.75                                       | 0.89 | 0.81 | 0.98 |
| The coefficient of heat resistance     | -                                           | 0.50 | 0.40 | 0.53 |
| The coefficient of crack resistance    | -                                           | 3.73 | 5.80 | 2.77 |
| Water saturation, %                   | 1.5…4.0                                     | 1.80 | 2.30 | 1.80 |

Analysis of the data presented in table 3 allows us to conclude that the composition of PMB No 2 without SWNTs, with a polymer content of 2.8 %, is characterized by increased sensitivity to temperature changes, which is typical for weakly bound structures. When introduced into the composition of PMB count, with a similar polymer content (composition No. 5), strong temperature-resistant structural bonds are formed, which give the composite a complex of improved properties. Thus, the improvement in the stability of the sensitivity of asphalt concrete based on PMB with SWNT to high temperature changes in heat resistance is 33% to low temperature changes is 52%. It is natural to assume that the preparation of asphalt concrete with a more rigid frame structure (type A, SMA) will be characterized by improved quality indicators.
4. Conclusion
Using the methods of optical density and refractive index, rational technological parameters for the preparation of the "carrier-carbon primary nanomaterial" system – ultrasonic dispersion for 2 minutes were established.

Microscopic light-field and dark-field imaging of polymer matrix film samples has shown that nanostructuring of interfacial layers occurs during modification of the polymer matrix, i.e., SWNTs affects the structure change. A more uniform distribution of the polymer in the plasticizing medium becomes possible.

Using the method of fluorescence microscopy, the effect of SWNTs on changes in the structure parameters of modified polymer-modified binders was established, which consists in reducing the polymer macromolecules and distributing them more evenly over the volume of the bituminous binder – structuring the composite system, which contributes to its stability.

The influence of the content of single-walled carbon nanotubes on the temperature range of the polymer performance of modified binders is established. Thus, the increase in the range of performance of the PMB, with the introduction of 0.0001% SWNTs, into its composition, was 43% in terms of resistance to high temperatures and 38 % in terms of resistance to low temperatures.

According to the method of Ya. N. Kovalev, the calculation of the required and required operating temperature range of asphalt concrete pavement was made, sufficient to ensure a given service life in specific natural conditions. It was found that the developed nanostructured composition of PMB with count 0.0001% exceeds the necessary requirements for the temperature range of the composite performance: by 14 % for the brittleness temperature and by 30 % for the softening temperature.

It is established that when preparing asphalt concrete based on PMB with SWNTs, stronger temperature-resistant structural bonds are formed, which give the composite a complex of improved properties. Thus, the improvement in the stability of the sensitivity of asphalt concrete based on PMB with SWNTs to high temperature changes in heat resistance is 33% to low temperature changes is 52%.

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