Review of Road Materials Self-healing: Problems and Perspective

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Abstract. Materials with the ability to restore their own functionality are a promising type of smart materials for road construction. The use of encapsulated functional modifiers is a common solution aimed at implementing self-healing technology. The paper presents general trends in the development of self-healing technology in the field of road materials. A mathematical interpretation of the destruction model of self-healing materials is proposed. The direction of technology development is proposed for the formulation of general requirements and quality indicators of self-healing materials for construction, characterizing the degree of their effectiveness depending on the conditions of use.

1. Introduction
The search for technological solutions to ensure the high reliability of building materials, which lead to an increase in the period of operation of the product or structure, is an actual direction in scientific research. Operating conditions vary over a wide range by type and intensity of factors. Road building materials are subject to the following factors: static or dynamic mechanical stresses, periodicity of climatic conditions (temperature, humidity, ultraviolet, oxygen, etc.), exposure to anthropogenic and natural chemical and biological agents. Usually, new technological solutions reduce the influence of some key factors, since the variability of the superposition of factors is high. The restoration of the performance of products and structures in practice occurs by repair and restoration work, which require economic costs.

For the development of the construction industry, materials with a unique set of properties, more functionality, less material consumption of production are required. A promising solution in the field of materials science to increase the durability of structures is the creation of “smart” materials [1, 2]. The formation of their own internal impacts, which ensures the maintenance of structure parameters at the required level, is a key condition for classifying the material as “smart”. “Reactions” of the material to a change in the structure parameters under the influence of operational factors of natural and / or artificial origin are provided at the material design stage.

For road construction, a promising type of smart materials are materials that have the ability to restore their own functionality in the design provided for by its purpose, which is usually called "self-healing" [3].

The development trend of scientific research in the field of self-healing technology shows (Figure 1) that in this direction there is an annual increase in the number of publications. At the same time, the share of work in the field of road materials by 2018 reached only 3.7%.

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In building materials science, self-healing technologies are used both in polymeric materials and in concrete based on hydraulic or organic binders. The self-healing mechanism is different for each type of building materials [4-9]. Traditional cement concrete, polymer concrete, and asphalt concrete can have their own potential for self-healing.

2. Results and discussion

The number of degradation mechanisms of the material is less than the variability of factors, this allows us to develop new technological solutions to eliminate the consequences of adverse changes in the structure of the material during the operational period. The formation and growth of cracks to critical sizes, after which they begin to spontaneously grow, is the result of the process of accumulation and development of defects (A. A. Griffiths in 1920 [10]). Placing a phase in the bulk of the material that is able to eliminate cracks and not be susceptible to or little susceptible to destruction under the conditions of operation of the material is a natural and fundamental solution. Since there are structural limitations, the reserve of the reducing phase must be designed at the material design stage and localized in areas with intense crack formation.

It is also important to clarify the term “self-healing” material. As a rule, the kinetics of changes in the structurally sensitive material parameter $F$ is described by the differential equation [11]:

\[
\frac{dF}{dt} = -k_t F^n,
\]

and his solution has the form:

\[
\frac{F(t)}{F(0)} = 1 - \sqrt[1 - n]{\frac{1 - n}{k_t F(0)^{1-n}}} t,
\]

where $F(0)$ is the value of the structurally sensitive parameter for the initial period of time; $n$, $k_t$ energy and kinetic constants; $t$ is time.

The kinetics of the change in the value of a structurally sensitive parameter for ordinary and self-healing material is presented in Figure 2.
An analysis of equation (1) shows that the rate of achievement of the initial critical state of the material, which is characterized by a decrease in the structurally sensitive parameter throughout the volume of the material and a local violation of its continuity (the formation of a volume defect, for example, micro- and macrocracks, but not magistral cracks), increases with an increase in the energy and kinetic constants. In this case, the durability of the materials is ensured at \( n \) and \( k_t \to 0 \).

The change in the structurally sensitive parameter during operation for ordinary material is described in accordance with formula (1), for which the energy and kinetic constants \( n_1 \) and \( k_{t1} \) characterize the intensity of the destruction process. The description of this process for a self-healing material is characterized by the presence of three stages of a structurally sensitive parameter change. The change in the structurally sensitive parameter \( F_1(t)/F(0) \) can be described using equation (1) in stage I. The self-healing stage of the material (stage II) occurs at the moment of the minimum acceptable value of the structurally sensitive parameter, which is determined during the design of the composition of the material. The rate of the self-healing process should be minimal, therefore, its description can be performed using a linear dependent:

\[
F_2(t)/F(0) = a \cdot (t - t_c) + F_1(t)/F(0),
\]

where \( a \) is the speed of the self-healing process, \( t_c \) is the time after which the critical value \( F(t_c) \) is reached and there is a need to restore the material.

The time \( t_c \) is optimal for the start of the self-healing process, the effectiveness of which is determined by the coefficient \( a > 0 \). After the completion of the self-healing stage, the change in the structurally sensitive parameter in stage III is also described by equation (1), for which the self-healing efficiency will be ensured for the values of the energy and kinetic constants \( n_2 \leq n_1 \) and \( k_{t2} \leq k_{t1} \).

The effectiveness of the self-healing technology is determined not only by the features of the recovery processes (speed, intensity, structure formation), but also by the features of the destruction of the reduced material, which are characterized by an energy and kinetic constant. Features of the effect on the change in the structurally sensitive parameter of the destruction of the material, due mainly to natural changes in the molecular structure and distribution of structural bonds in the matrix, must be taken during the design process of self-healing material.

The intensity of destruction from this factor is reflected in the value of the energy constant \( n \), and the intensity of destructive from mechanical operational influences is reflected in the value of the kinetic constant \( k_t \). In the absence of external mechanical influences \( k_t = 0 \), even in the presence of internal stresses in the material \( n \neq 0 \), there will be no additional energy for the formation of defects in the material and the values of the structurally sensitive parameter will not change.

The self-healing process is accompanied by a change in the structure parameters, the formation of new structural elements, the redistribution of structural bonds, and a change in their strength, which should lead to the formation of a structure whose destruction rate is less than the destruction rate of the primary structure of the material. The calculation of increasing the life of the material due to self-healing is evaluated not at the moment of reaching the initial critical state of the material (p. A and C.}

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**Figure 2.** Schematic representation of the kinetics of changes in a structurally sensitive parameter: 1 - ordinary material; 2 - self-healing material
in Figure 2), but at the moment of reaching the minimum value of $F(t)/F(0)$ or $F_2(t)/F(0)$, respectively, i.e., $D$ and $D'$ in Figure 2.

Self-healing in a material extends to a separate category of defects that this process can influence. These include defects with parameters not exceeding the value, the corresponding critical state when the process of their further development acquires an avalanche-like character.

In this regard, a “self-healing” material is a material capable of initiating the process of eliminating structural defects, the flow rate of which exceeds the rate of their spontaneous growth, and the resistance to operational conditions of the formed structure is equal to or greater than the resistance of ordinary material.

Thus, the development of solutions for the implementation of this principle will increase the life of materials, which is an urgent task of materials science, including in the direction of road construction. Traditionally, cement stone occupies from 10 to 30% of the volume in the composition of concrete, of which 20...30% remain not hydrated (clinker stock). The amount of clinker stock in the volume of concrete depends on the degree of dispersion of Portland cement and the water-cement ratio. During the operation of structures made of such concrete under conditions of natural cracking, water can penetrate into the material, which promotes Portland cement (clinker stock) hydration, and the products of this reaction can fill cracks and other defects, which can be described as a self-healing structure [12].

The difficulty of predicting the access of the required amount of water to start the process of hydration for the formation of reaction products sufficient to stop the defect is the main problem of implementing the technology based on the hydration potential of cement grains inside the concrete. However, the features of the hydration potential can be used in the production of asphalt concrete using a Portland cement filler. In this case, water penetrating under the bitumen films promotes the start of the hydration process and the formation of hydrosilicate crystals that penetrate the film, increase adhesion and prevent its peeling [13].

Developments using alkaliphilic endospore-forming bacteria [14-20], which starts the process of biomineralization of concrete with subsequent precipitation of calcite [21], are common solutions for creating self-healing material. Bacteria of the genus bacillus are used to provide a regenerative process, which is added to the concrete together with the calcium salt of lactic acid (calcium lactate) and nutritional supplements [22-24]. Bacteria begin their life after interacting with water, consume oxygen, eating calcium lactate, which is subsequently converted to calcium carbonate. Thus, soluble calcium lactate is converted to insoluble calcite, which helps to stop cracks and pores inside the material, preventing moisture from penetrating and increasing frost resistance [25]. At the same time, the corrosion resistance of reinforcing elements in concrete increases when bacteria consume oxygen [26].

Bacterial carriers are used to prevent premature activation of the reproduction process, which concentrate spores and prevent their contact with water during the preparation of the concrete mixture. In [27, 28], the bacteria carrier is expanded clay aggregate, which is saturated together with a nutrient solution and added to the concrete mix. However, the low strength of the aggregate used leads to a decrease in the compressive strength of concrete to 50%.

An alternative method of encapsulating bacterial spores with nutrients is a hydrogel, which has the ability to absorb and retain moisture [29]. The encapsulation method presented allowed using calcium carbonate to stop cracks up to 0.5 mm wide and reduce water permeability by 68%.

The most widespread methods of encapsulating reducing agents are various capsules, which are made on the basis of polymeric compounds that form capsules during curing [30-34]. The decrease in the physicomechanical properties of concrete is the main problem in the use of capsules with a reducing agent. At the moment, there is not enough data confirming the improvement of physical and mechanical properties of the material after recovery using various techniques for the synthesis of encapsulated modifiers.

The implementation of self-healing technology in asphalt concrete is associated with its structural features and thermoplastic properties. The increase in the strength characteristics of asphalt concrete in accordance with regulatory documents does not ensure its durability in operating conditions, which is associated with the peculiarity of the properties of bitumen [35-37]. An increase in the tensile strength
of asphalt concrete at high temperatures leads to an increase in the tensile strength at low temperatures, which negatively affects its fragility.

The balance between maximum strength at positive temperatures and minimum brittleness is one of the conditions for ensuring durability. Durability is the maximum possible lifetime of the asphalt concrete pavement, in which the serviceability is maintained and repair or restoration is not carried out, possibly in the case when the state of the material structure does not change or the ability to respond to external factors without the formation of defects is preserved. Irreversible processes occur leading to the formation of defects and disruption of the structure in asphalt concrete during operation of the pavement under the influence of weather and climate factors and traffic load.

The durability of asphalt concrete is inversely proportional to the speed and intensity of the course of destructive processes. Thus, to increase the service life of the asphalt concrete pavement, solutions are needed to ensure the duration of the state of the structure of asphalt concrete without defects due to giving it unique properties, independently restore the integrity of the composite and its ability to resist influencing factors.

Various types of impregnators are used to extend the service life of asphalt concrete pavement during the operation phase in order to prevent the development of primary defects [38-42]. Impregnating compositions after surface treatment of the asphalt concrete coating diffuses into the material, restoring the molecular composition of the asphalt binder, which leads to a decrease in stiffness [43, 44].

Rejuvenators, low molecular weight organic compounds, are the main component of such impregnating compounds that dissolve in bitumen, compensate for the loss of malleable fractions during aging during operation [45-52].

However, the penetrating ability of anti-aging compositions depends on rheological properties and does not exceed 20 mm [53], therefore, the restoration effect extends only to the surface layers of the coating. Stopping traffic to use asphalt restoration technology using impregnating rejuvenators is also a drawback.

The use of functional “containers” with a rejuvenating agent or modifier, in the form of capsules or hollow fibers, allows you to get rid of these drawbacks. The implementation of this solution will allow asphalt concrete to independently respond to structural changes in order to restore the functional state during operation. Such self-healing material is classified as “smart.”

It should be noted that bitumen has its own potential for self-healing, which depends on both physical and chemical properties [54]. Self-healing of a binder in asphalt concrete occurs in the absence of a dynamic effect, which is aimed at restoring fatigue damage in the material. The degree of recovery is determined by the relaxation time [54-56].

The result of the self-healing process is the restoration of the integrity of the structure of physicochemical bonds by means of wetting the surfaces of cracks, diffusion of molecules between surfaces and arbitrary scattering of molecules, providing hardening [57, 58]. In bitumen, self-healing can occur at the molecular level through reversible hydrogen bonds with the formation of new cross-links and chains [58-60] through ditopic and tritopic molecules [60]. The intensity of the recovery process in bitumen depends on the temperature conditions in which it will be in the period of rest from repeated loads. The recovery process of bitumen proceeds more intensively at a higher temperature. This feature of bitumen is used in the recycling of asphalt concrete.

The self-healing potential of bitumen is used in the technology of induction recovery of asphalt concrete pavement, which was developed at the Delft University of Technology [62]. In accordance with [63-65], 5...7% of the metal fiber is introduced into asphalt concrete, which, using a special induction installation, is exposed to a high-frequency alternating electromagnetic field and is heated into the pavement. Bitumen around heated metal fibers is melted above the softening temperature, recovery occurs and defects in the binder are eliminated [55, 66, 67]. The healing process can be repeated at least five times with an equal degree of recovery (Figure 3).

The temperature gradient across the thickness of the pavement is a drawback of such a technology that occurs during induction heating (Figure 4).
The temperature difference on the surface and in the volume at a depth of 80 mm can reach more than 90 °C with an increase in the time of induction exposure with a power of 8.3 kW and a frequency of 123 kHz. At the maximum depth, the temperature of the material is optimal for recovery, which is equal to the softening temperature of bitumen − 47.5 °C, which is not achieved [65].

Thus, the use of induction heating to restore the entire thickness of the pavement is currently impossible. The need to develop special induction equipment increases the cost of introducing technology.

The uniformity of the self-healing process in the volume of the material can be achieved using encapsulated functional agents, which are added to the composition of the asphalt mix together with other components. However, the use of encapsulated modifiers or bacteria in asphalt concrete is associated with a number of technological features of the preparation of the asphalt mix.

Capsules with a reducing agent should be heat-resistant at temperatures of asphalt-concrete mixture preparation - from 140 to 185 °C and resistant load compaction of the asphalt-concrete mixture.

There are various technologies for the production of microcapsules containing a reducing agent for asphalt concrete, which differ in the initial components, technological process, and product properties.

In work [68], two-stage coacervation technology is used for the manufacture of microcapsules based on a methanol-melamine-formaldehyde-modified methanol prepolymer. The technology of using melamine formaldehyde in the manufacture of capsules with an anti-aging agent is environmentally unsafe [69].

In [70], the options for obtaining the capsule shell from cellulose and polymer for an emulsified reducing agent in water were considered.

The authors of [71] proposed a three-stage method for producing microcapsules, in which at the first stage styrene-maleic anhydride and water are mixed for 2 hours at a temperature of 50 °C, then NaOH is added to pH 10 and a reducing agent is added. In the second stage, the resulting emulsion and methanol-melamine-formaldehyde are mixed while heating to 80 °C with a rate of 2 °C/min. In the third stage, the emulsion is kept at 80 °C for 2 hours, after which the resulting capsules are filtered and dried in a vacuum oven.

In [72], a multistage encapsulation technology of a reducing agent was proposed, in which the reducing agent emulsified in water is coated with a urea and formaldehyde coating.

The most technologically simple way to obtain microcapsules was proposed by the authors [73]. Sodium alginate (CaH2O3Na), which is the sodium salt of alginic acid, is used to make calcium alginate capsules. Sodium alginate is poorly wetted by water, individual particles of alginate powder are actively aggregated in water with the formation of agglomerates. Sodium alginate is added to water and mixed for 2 minutes using an overhead mixer with a drive rotation speed of at least 2000 rpm [73]. The destruction of agglomerates occurs in the process by high-speed mixing and a colloidal solution is formed. A reducing agent is added to the resulting suspension and mixed for 2 minutes. The resulting emulsion is divided into capsules through a dropping funnel with an outlet of 0.71 cm. The division is
carried out in an aqueous solution of calcium chloride. After filtering and washing, the obtained capsules were dried in a dry oven at a temperature of 40 °C for 12 hours.

A suspension of sodium alginate is added dropwise to the calcium chloride solution, where gelation of the alginate occurs (at pH<4) in the next stage of capsule preparation. The mechanism of the formation of alginate gels consists of joint binding of calcium ions between macromolecule ribbons located in one line, which have pores or cavities of 0.19..0.20 nm in size corresponding to the diameter of Ca^{2+} ion [74]. Intensive gelation occurs when pores are filled with calcium ions; crosslinking of macromolecules through substituted sodium cations Na⁺ occurs. The separated drops of an alginate gel with an emulsified reducing agent are dried after washing with distilled water. Evaporation of excess moisture from the gel and a decrease in volume occurs during the drying process, the emulsion breaks down inside an individual alginate gel particle, calcium-alginate capsules with a reducing agent are formed.

Rejuvenators, organic oils with different molecular weights are used as a reducing agent by the authors of all the works reviewed. The mechanism of action of such a reducing agent in asphalt concrete is the dissolution of bitumen components aged during operation and local reduction of fragility.

The result of encapsulation technologies is the production of micro containers containing a reducing agent, which differ in the initial components and properties of the resulting capsules. The main characteristics of such containers with various shapes are presented in table 1.

Capsules obtained by various methods have sizes from 10 μm to 3 mm, in which the encapsulated reducing agent is predominantly vegetable oils [68, 69, 72-80]. Industrial rejuvenator is an Alternative variant of the reducing agent, which is a mixture of low molecular weight compounds and oils [70-72, 75].

As a rule, the capsule shape is spherical or ellipsoidal, which facilitates their use in the process of mixing with other components of the asphalt mix. The technology has a significant drawback if the container for the reducing agent is fiber: premature destruction of the containers during mixing and during operation.

The highest content of the reducing agent in the capsules is achieved in the shells of calcium alginate, which is explained by the simplicity of the preparation technology and a wide range of variable dosages of the components.

Capsules produced by various technologies must have a strength that ensures their integrity during the preparation of the asphalt mix and its compaction.

The quality of the self-healing technology depends on the technological properties of the capsules and the restoring properties of the encapsulated agent. A single methodology for controlling the ability of a material to self-healing does not exist at present. There are no criteria for assessing the ability of a material to independently respond to external conditions and eliminate an adverse effect on the properties or structure of the material.

In [81], quality criteria were proposed that reflect the effectiveness of a material with self-healing ability: the degree of restoration of the state of the structure; rate of restoration of the state of the structure; the durability of the restored structure and the timeliness of initiating the recovery process. However, a large number of empirical studies are necessary to select property indicators that characterize each of the quality criteria.

Quality indicators, which reflect the self-healing ability of asphalt concrete, are presented in table 2.

| № | Capsule material | Form | Size | Reducing agent (RA) | Volume RA | Reference |
|---|------------------|------|------|---------------------|-----------|-----------|
| 1 | Methanol- melamine-formaldehyde prepolymer | Sphere | Diameter – 100 μm | Sunflower oil | – | [68, 69] |
Table 2. Self-healing performance indicators of asphalt concrete

| №  | Indicator            | Condition for healing | Effect             | Reducing agent                                                                 | Reference |
|----|----------------------|-----------------------|--------------------|--------------------------------------------------------------------------------|-----------|
| 1  |                      |                       |                    |                                                                                |           |
| 2  | 3-point bending      | 6…192                 | 20±2               | up to 53 %                                                                     | [76]      |
| 3  | bending              | 3                     | 20±2               | 50 %                                                                           | [78]      |
| 4  | strength             | 3                     | 20±2               | 30 %                                                                           | [78]      |
| 5  |                      | 3                     | 20±2               | 24 %                                                                           | [78]      |
| 6  | Tensility            | 6…216                 | 20±2               | 54…95 %                                                                        | [82]      |
| 7  |                      | 5…216                 | 20±2               | 20…55 %                                                                        | [84]      |
| 8  | Stiffness            | 0.5…1.5               | 21±1               | 15…90 %                                                                        | [83]      |
| 9  |                      | 4                     | 21±1               | 80…100 %                                                                       | [83]      |
| 10 | Tensile strength     | 6…48                  | 21±1               | up to 80 %                                                                      | [83]      |
| 11 | Crack resistance     | 20                    | 23±2               | 14…19 %                                                                        | [86]      |
| 12 |                      | 20                    | 20±2               | –                                                                               |           |

2  Sepiolite  Sphere  –  Industrial rejuvenator  40 %  [70]
3  Polymer    Sphere  Diameter – 1.00…1.30 мм  Industrial rejuvenator  80 %  [70]
4  Methanol-melamine-formaldehyde prepolymer  Sphere  Diameter – 10…20 мкм  Industrial rejuvenator  50 %  [70]
5  Methanol-melamine-formaldehyde prepolymer  Sphere  Diameter – 57.5 мкм  Oil containing polar epoxy groups  80 %  [72]
6  Calcium alginate  Sphere  Diameter – 2.90 мм  Sunflower oil  48 %  [73]
7  Calcium alginate  Sphere  Diameter – 1.95 мм  A mixture of rejuvenator with an olefin-maleic anhydride polymer solution  56 %  [75]
8  Calcium alginate  Sphere  Diameter – 2.90 мм  Sunflower oil  46 %  [76]
9  Calcium alginate  Cylinder  Diameter – 0.5 мм  Sunflower oil  70 %  [77, 78]
10 Calcium alginate  Ellipsoid  Diameter – 0.98…1.74 мм  Sunflower oil  60 %  [79]
11 Calcium alginate  Sphere  Diameter – 1.95…2.45 мм  Sunflower oil  90 %  [79]
12 Calcium alginate  Sphere  Diameter – 2.5 мм  Sunflower oil  94 %  [80]
| Fatigue life | 20 | 20±2 | A mixture of a reunitor with a polymer solution of olefin-maleic anhydride [86] |
| Bending strength | 72 | 30±2 | 50…89% | Rejuvenator [87] |

All proposed methods for assessing the self-healing ability of materials are reduced to calculating the coefficient of relative change in the measured indicator:

\[ HL = \frac{X_h}{X_a} \]

where \( X_0 \) and \( X_5 \) are the index of material properties before and after self-healing, respectively.

In accordance with [81], this coefficient can be assigned to the category of indicators that reflect the degree of restoration of the state of the structure, which is insufficient for an objective assessment of technological solutions.

Fatigue life, which reflects the dynamic loading pattern in the pavement, is used as an indicator for assessing the self-healing effect of the material only in [86]. In other cases [75, 76, 78, 82-85, 87], the authors use property indicators characterizing the operational state of the material at the time of failure, when the defectiveness of the structure is ultimate — the structure is destroyed. However, in the pavement, the initiation of a self-healing process should occur at the stage preceding the destruction of the structure.

There is no unified evaluation system, which allows you to objectively compare the effectiveness of microcapsules with a reducing agent produced by various technologies and evaluate the effects of prescription and technological factors on the self-healing process and its result. Thus, it is necessary to improve the technological aspects to obtain effective capsules and develop methods for evaluating the effectiveness of technologies: the degree of change in the defectiveness of the structure; the rate of its change during recovery; the kinetics of the formation of defects in the structure after the self-healing process. A key task in the field of self-healing technology is to establish the defectiveness of the material at which the start of the self-healing process is the most effective.

3. Conclusion

An analysis of the scientific and technical literature shows a growing interest on the part of researchers and technologists in the field of technology of self-healing materials.

Self-healing materials are materials that have the ability to independently initiate processes of restoration of structural parameters without an additional energy cost, the rate of which exceeds the rate of formation of defects.

Existing technical solutions allow you to get capsules containing a reducing agent up to 90...94% of the total volume.

It is necessary to formulate general requirements and quality indicators for self-healing materials characterizing the degree of their effectiveness depending on the conditions of use.

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