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IMPROVEMENT OF RECEIVING TECHNOLOGIES DISPERSED-FILLED LIGHT-REFLECTIVE MATERIALS

05.16.09 - Materials science (in mechanical engineering)

AUTOABSTRACT
thesis for the degree
candidate of technical sciences

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The work was carried out on the department «Materials, welding and production safety» of the FPBEA NE the «Kazan National Research Technical University named after A.N. Tupolev – KAI» (KNRTU-KAI)

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The thesis can be found in the library of the Federal State University OF THE RUSSIAN Academy of Sciences.

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GENERAL PERFORMANCE

Relevance of the study topic. The increase in reliability and durability of products, structures and complex technical systems operated in various climatic conditions under the influence of high and low temperatures, static, dynamic and vibration loads, environmental ingredients and many other factors is inextricably linked to the improvement of traditional and the development of new materials of various functional applications and technologies for their production.

In many areas of technology, including in machine building, luminous-growing materials (RM) are widely used in the form of dispersed-filled films and coveredvarious structures, characterized by the ability to return the stream of light falling on them back to the direction of the light source. RM is widely used in machine building for marking various vehicles, including heavy-duty cars, hydraulic presses, crane beams, bridge cranes, as well as elements of measuring systems in many industries. There are various technologies for obtaining dispersed-filled RM, differing in reflective elements, initial components and structure. The efficiency of RM I is determined, first of all, by the type of light-reflectors, composition, structure and technology of reception, the type of radiation source (wavelength or range of its change).

The leading position among the world manufacturers of RM is occupied by the company "3M" (USA), which produces a wide range of different types of film materials using microspherical and angle light-reflectors. It should be noted, however, that such SVMs are of high value. In addition, the technology of obtaining SIUD with angle reflectors is difficult, it requires a complex of expensive equipment, while the technology of manufacturing RM with the use of microspheres is simpler, technological and economically expedient. Therefore, studies aimed at developing the technological processes of receiving RM c with the given light-growing properties of other operational properties, establishing the features and patterns of their change from nature, content, ratio and combination of components, technology and regime parameters of formation, are relevant.

Degree of development of the topic. The research is carried out on the basis of fundamental experimental and theoretical research in the field of development of compositions and technology for the production of dispersed-filled |RM multifunctional purpose, carried out in the works of such domestic and foreign scientists as Baryshnikov N.V., Karasik V.E., Ivanov L.A., Slugin V.A., Kizevetter D.V., Malyugin V.I., Kiselev N.N., Molochina L.A., Filin S.A., Belkin N. Yu., Narusbek.A., Potapova N.I., Tsvetkov A.D., Yakovlev A.D., Maslyakovsky L.N., Vereshchagin I.P., Belotserkovsky M.A., Gotz V.A., Krakovich G.A., Bely B.A., Dovglyak V.A., Yurkevich O. R., Belyakov Yu. A., Muslimov E.R., Pryakhin Yu., Garipov R.R., Galimov E.R., Fedyaev V.L., Belayl L. K., Borden T.R., Grunzingeri R.E., Solméno S., Crozinyani B., Di Porto P., Gerard A., Barch J. Muller B., Pot W., Lamburg L. and many others.

The main task in the development of technologies for obtaining RM various composition and a given structure is the formation of a certain geometry of the elements of the microrelief surface, which provides the necessary luminous-turning properties. Improvement of technologies for the formation of RM with pre-defined and regulated properties through the use of components of domestic production will provide a significant technical, economic and social effect.

The purpose of the work is the development of compositions and technologies for obtaining dispersed-filled light-growing materials of various structural forms of film and coatings with increased operational and technological properties.

In accordance with the goal set, the following tasks were solved:

1. Based on the analysis of existing production technologies, select the initial components for the formation of dispersed-filled microspheres of light-producing materials of various composition in the form of films and coatings, to define their main characteristics (refractive index, dispersion, thickness, etc.).

2. Improve the experimental installation to improve the accuracy of the measurement of the reflection indicator of the RM in a wide range of angles of drop of the beam.

3. Investigate the influence of the nature, composition, ratio, combination, optical and other characteristics of the original components on the change in the intensity of light-reversal and the operational properties of flexible multilayer thin-film RM based on glass microspheres and functional layers. To develop the technology of obtaining multilayer RM, providing maximum luminous-turning properties.

4. Investigate the effect of the characteristics of the original components on the change in the intensity of light-ovation and the complex of operational properties filled with microspheres of coatings based on polymer powder compositions. To reveal conditions of manifestation of maximum effect of cathodioptric light-binding in multilayer coverings at change of thickness of functional layers and dispersion of microspheres.

5. Using the results of the research, develop practical recommendations aimed at developing technologies for obtaining RM characteristics that meet the operating conditions.

Objects of interest. Microwave tubes in the form of films and coatings filled with glass microspherical reflectors; technological processes and regime parameters of their formation.

Methodology and methodology of research. The methodological basis is a systematic approach to the development of technologies for obtaining the MUs of various composition and structure. In the course of the thesis, standard and developed methods for the study of the structure and properties of the RM are chosen.

The scientific novelty of the work is the following:

1.Patterns of influence of nature, composition, combination, optical and other characteristics of initial components, as well as regime parameters of formation on change of light-changing operational properties of RM on the basis of glass microspheres are Established and scientifically justified.

2. It was found that the achievement of the required level of operational, including light-reflective properties of flexible multilayer thin-film RM is ensured by the optimization of the composition and the use of technological techniques to improve the structure of the RM.

3. The regularities of the influence of nature, composition, ratio, characteristics of initial components and regime parameters of formation on the change of properties of RM in the form of coatings based on epoxy, polyester and epoxy-polyester powder compositions filled with microspheres are established and scientifically justified.

Theoretical and practical significance of the work.

1. In the scientifically and experimentally justified choice of the initial componentsof various structures that can increase the level of their technological and operational properties.

2. The experimental installation has been improved to improve the accuracy of measuring the intensity of light-loop samples of RM in a wide range of beam-drop angles that meet operational requirements.

3. The technological processes of obtaining a RM with the use of microsphericallight-reflectors have been developed. Optimal combinations and component ratios, technological features of multilayer structure formation are determined by operational properties.

4. The efficiency of development of RM on the basis of epoxy, polyester, epoxy-polyester powder compositions and glass microspheres is shown. Variants of structures and modes parameters of formation of the RM are developed, ensuring increase of intensity of their light-saturation and other technical properties.

5. Scientific recommendations on development of highly effective and accessible technologies of obtaining of various composition and a given structure using components of domestic manufacture are proposed.

Main provisions for the defence:

- a set of results of experimental studies on the development of formulations and regime parameters of technological processes for the production of flexible multilayer RM s on the basis of microspherical light-reflectors;
- a set of results of experimental researches on development of compositions and modes parameters of obtaining of the RM on the basis of filled glass microspheres of polymeric powder coatings;
- scientifically based technical solutions for formation of various composition and structure, providing a given level of intensity of light-ovation and operational properties.

The correspondence of the thesis with the passport of a scientific specialty.

Thesis work on its purpose, solved problems, content, methods of research, scientific novelty, theoretical and practical significance corresponds to the passport of specialty 05.16.09 - Materials science (in machine building) on the following items:

- p.5. The influence of the regimes of technological impacts in the production of materials on their structure. Optimization of the technology of obtaining materials of the given structure and properties;
- p.9. Development of coatings for various purposes (hardening, wear-resistant and other) and methods of their quality management.

Publications. According to the materials of the thesis, 17 works were published, including 2 publications from the list of publications defined by the higher Attestation Commission of the Russian Federation, 1 article is included in the international citation base Scopus, 5 articles 9 theses of reportsin the materials of all-

Russian and international conferences, a patent of the Russian Federation for invention was received.

Реализация работы. Полученные результаты диссертационной работы используютсяв учебном процессе кафедры «Материаловедение, сварка и производственная безопасность» ФГБОУ КНИТУ-КАИ им. А.Н. Туполева.

Implementation of the work. The results of the thesis are used in the educational process of the Department «Materials science, welding and production safety» FPBEA KNITU-KAI named after A.N. Tupolev.

The author's personal contribution is to select and base the original components of the RM, prepare samples for testing, conduct experimental studies, process and analyze the results obtained. The purpose and objectives of the study are drawn up, discussions are held and the interpretation of the results is carried out jointly with the scientific supervisor.

The reliability and validity of the results of the research are confirmed by their reproducibility and consistency of the experimental results obtained using modern equipment, the use of generally accepted and special techniques, as well as the correlation of the obtained data with the results of the studies performed by other vehicles on the topic of dissertational work.

Work approbation. The main provisions of the thesis were reported and discussed at all-Russian and international conferences: VIII International scientific and technical conference «Innovative machine-building technologies, equipment and materials» (Kazan); International Youth Scientific Conference «Tupolev readings (School of Young scientists)» (Kazan); International conference «Energy saving. Science and education» (Naberezhnye Chelny); All-Russian scientific and practical conference with international participation «New technologies, materials and equipment of the Russian aerospace industry» (Kazan); International scientific and practical conference «Scientific potential of Youth and Technical progress» (St. Petersburg).

The work was carried out within the framework of the State task of the Ministry of Education and Science of the Russian Federation. Assignment №9.3236.2017/4.6, 31.05.2017.

The structure and scope of the dissertation are determined in accordance with the need to solve the assigned goal and tasks. The work consists of introduction, four chapters, main conclusions, a list of literature of 147 titles and annexs. The work is presented on 125 pages of text, including 68 figures and 9 tables.

The author expresses gratitude to the scientific leader - professor, doctor of technical sciences Galimov E.R. for valuable advice and support, as well as employees of the departments «Materials science, welding and production safety», «Opticoelectronic systems» KNITU (KAI) named after A.N. Tupolev for help in the discussion of the results of experimental studies.

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THE MAIN CONTENT OF THE WORK

The introduction justified the relevance of the theme of its development, the choice of the subject of the thesis study was argued, the purpose and tasks of the work were formulated, the choice of objects and methods of research was justified, the main provisions made for protection were given, scientific novelty, theoretical and practical value, structure and scope of dissertational work was shown, the content of the thesis was reviewed.

In the first chapter the literary review on the topic of dissertational research is carried out, in which the main optical characteristics of materials, types of substrates and light-rotating elements in the form of microspheric reflectors, as well as the existing technologies of obtaining of the RM of different composition and structure are considered. The classification of polymer powder compositions and existing technological processes of coating production on their basis is given. Based on the analytical review, well-founded generalizations and conclusions are made. It is shown that studying the peculiarities of the influence of composition, structure and technologies of obtaining is an actual task, the solution of which will contribute to the development of the civil engineering with the given tasks and increase the efficiency of their application in many branches of industry, including in engineering.

In the second chapter the description of the basic physical-chemical and optical characteristics of the initial components for the production of light-reflective materials in the form of films and coatings, methods of preparation of samples and methods of experimental researches of initial components and compositions on their basis is given. When forming flexible multilayer RM as light-reflective materials, glass microspheres of the brand «SMSH» (TU 6-48-91-92) of various dispersion are chosen. As functional layers are used: Reflecting layer in the form of foil aluminum, aluminum foil, aluminum foil FG; Intermediate layer in the form of composition on the based on transparent varnishes AK-593 (TU 6-10-1053-75), HB-784 (GOST 7313-75) and aluminum powder PAP-1 (GOST 5494-95) at various their ratios, composition on the basis of «Metallic silver 9007» and aluminum powder; binding layer in the form of transparent polyester tape; Separating layer in the form of a grid on the basis of synthetic threads (GOST 4403-91); protective layer on the basis of polyethylene terephthalate film (GOST 24234-80). Epoxy powder compositions PEP-219 and PEP-91, polyester P-PE-1130 and epoxy-polyester Pigma P-201 were used for obtaining coatings. Dispersion of microspheres was determined on the diffraction-laser analyzer «Masterizer 3000 Aero S» according to GOST 31993-20131 and the city methode according to GOST 4403-91, the defect of the microsphere structure of the RM was estimated using the inverted annotation microscope of the brand «Axiovert 200 MAT» according to GOST R 53172-2008, the parameters of the initial components of the RM according to GOST 28869-90. For the study of light-developing properties of RM in the form of films and coatings, an installation developed at the Department «Materials science, welding and production safety» KNITU (KAI) named after A.N. Tupolev. Operational characteristics of films and coatings were determined by standard methods: Adhesion according to GOST 15140-78, covering according to GOST 8784-75; Appearance according to GOST 9.407-84, weather-resistant according to GOST 6992-68; Resistant in liquid corrosive media according to GOST 9.083-78, light resistance according to GOST 9.045-75.

In the third chapter presents the results of studies of the retroreflective properties of flexible multilayer CBMs in the form of thin films using microspheres and functional layers (reflective, intermediate, binder, separating, protective). In the figure 1 as an example the data on changes of the reflection indicator of reference sample «3M» and samples of RM obtained from the use of selected components in their various combinations and ratios are presented.

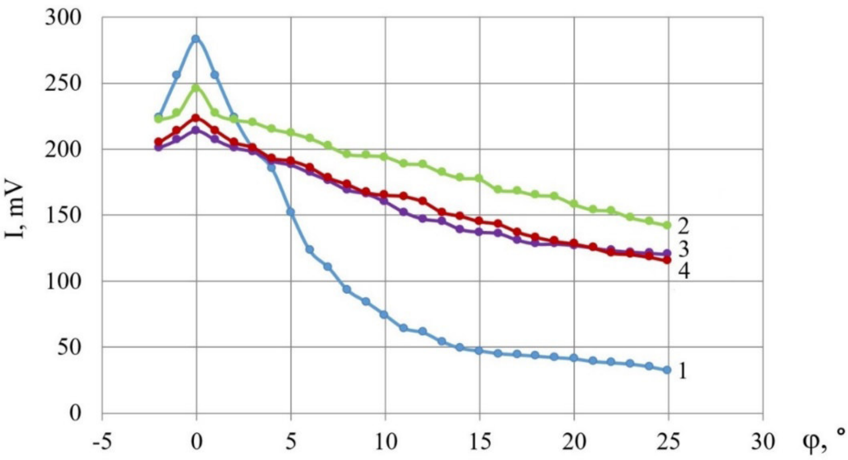


Figure 1 – Change of the reflection intensity of the RM with microspheres of dispersion 48-56 µm from the angle of the beam drop: 1 – sample "3M", 2 – sample with reflective layer - foil aluminum, intermediate layer - varnish AK-593 + aluminum powder in ratio 99.66:0.33; 3 - a sample with a reflective layer - foil aluminum, intermediate layer - AK-593 varnish + aluminum powder in a ratio of 68.35:0.23; 4 - a sample with a reflective layer - aluminum FG foil, an intermediate layer - AK-593 varnish + aluminum powder in a ratio of 99.66:0.33

When comparing sample 2 with a reflective layer, foil aluminum and an intermediate layer based on AK-593 lacquer and aluminum powder in a component ratio of 99.66:0.33% with sample 3 with the same reflective and intermediate layers, but with a component ratio of 68.35:0.23%, it can be noted, that sample 2 has a reflection intensity higher than sample 3 over the entire range of laser beam fall angles. In addition, comparing samples with the same dispersion of microspheres 48-56 microns and the same intermediate layer - AK-593 varnish + aluminum powder with a ratio of 99.66:0.33%, but with different reflective layers (foil aluminum and aluminum foil), one can conclude, That in sample 2 with reflecting layer in the form of foil aluminum reflection intensity is noticeably higher than in sample 4.The influence of quantitative thickness of the intermediate layer on the change in intensity of light-ovation has been studied. Figure 2b shows the results of the samples with different thickness and quantity of the intermediate layer based on the transparent lacquer of HB-784 as an example.

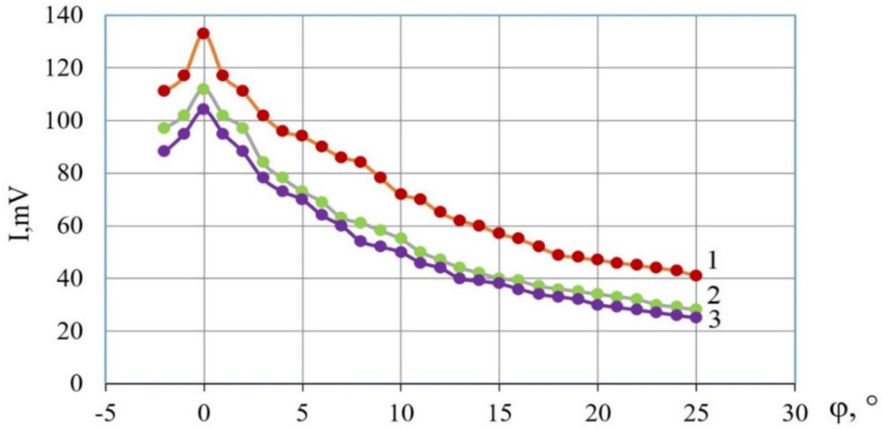
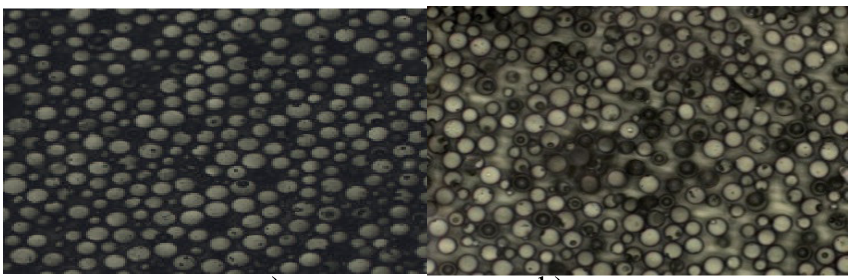


Figure 2– Change of the intensity of the reflection of the SVM from the angle of the laser beam falling.
Curves 1, 2, 3 are samples with one, two and three layers of HB-734 lacquer, microspheres of dispersion 48-56 microns and reflecting layer in the form of foil aluminum

Figure 3 shows examples of light-rotating foil-based aluminum FG foil samples with intermediate and non-intermediate layers as an example.



a) b)

Figure 3 – Reflective RM images - Aluminum FG foil:
A) intermediate layer sample - clear AK-593 varnish + aluminum powder in ratio 99.66:0.33%, b) sample without intermediate layer (increase of 100x)

The dependence of the intensity of the reflection of the RM on the type of the reflecting layer was evaluated by constructing the reflection indicator diagrams from the degree of dispersion of microspheres with a refractive index of 1.54 (figure 4).

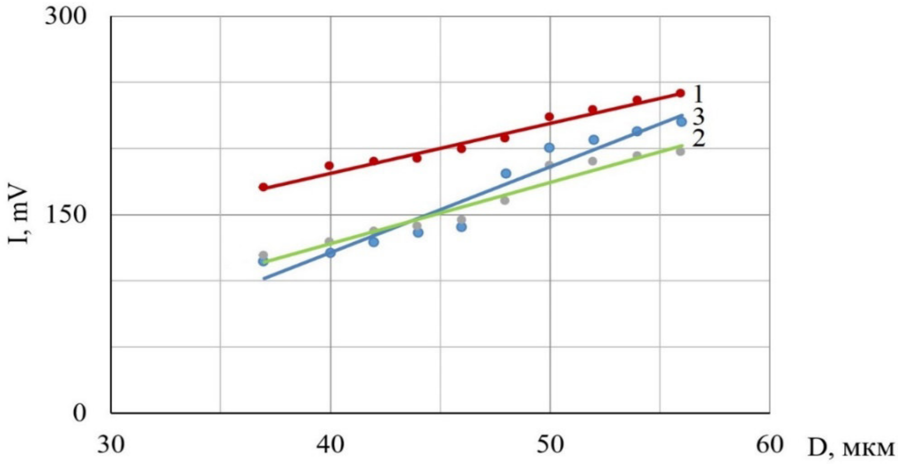


Figure 4 – Change of the intensity of the reflection of the RM from the dispersion of microspheres: 1, 2, 3 - samples with reflective layers - foil aluminum, aluminum Foil, foil aluminum FG

From the experimental data presented, it can be seen that the maximum amount of reflection intensity is manifested for the microwave with the same dispersion of microspheres when used as a reflection layer in the form of foil aluminum. For aluminum foil and aluminum FG aluminum foil, the reflection intensity values are noticeably lower and have approximately the same values. The observed effect is due to the higher quality of the surface of foil aluminum and the purity of treatment. Figure 5 illustrates the variation in the intensity of the reflection of the RM at different volumes of defective microspheres.

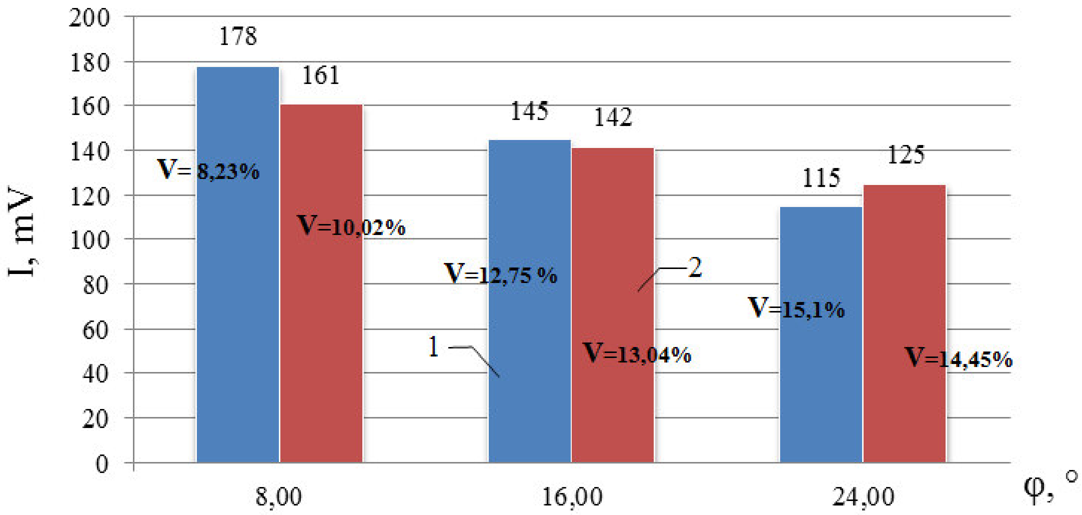


Figure 5 – Change in the intensity of reflection of the CBM from the angle of impact of the laser beam at different volumetric fraction of defective microspheres: reflective layer - aluminium foil oculphere 48-56 um: 1 –593 + aluminium powder ratio 99.66:0.33 %.2 – sample without intermediate layer

A new technology has been developed for producing a flexible multilayer thin-film retro-reflective material (RF patent 2660048) containing an upper protective layer with an image sealing a layer in the form of a mesh with microspheres, partly in depth into a polymeric binder layer (Figure 6).

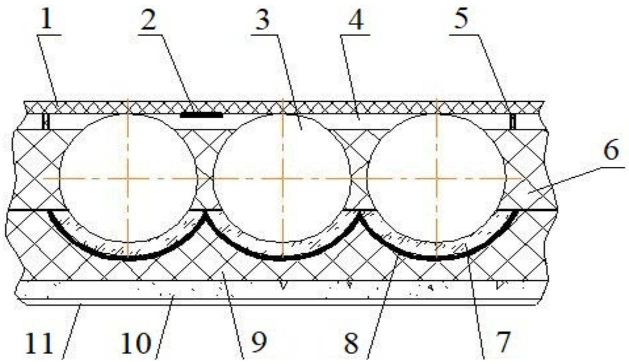


Figure 6– CBM structure:
1 – protective layer; 2 – image;
3 – microspheres; 4 – air;
5 – sealing layer, 6 - binding layer, 7 - focusing layer; 8 –Metallized surface; 9 – mounting layer; 10 – adhesive paper; 11 – protective film

The upper protective and binding layers are connected to each other so that an air layer is formed between the back of the protective layer and above the surface of the microspheres. On the rear hemisphere of the microspheres is a focusing layer with a metallized reflective surface. The focusing layer is a transparent polymer film with a refractive index equal to the refractive index of microspheres.

The fourth chapter, the retroreflective properties are studied, the compositions are determined, and the technology for producing RM with microspheres is developed, in which coatings based on thermosetting powder compositions are used as the base binding layer.

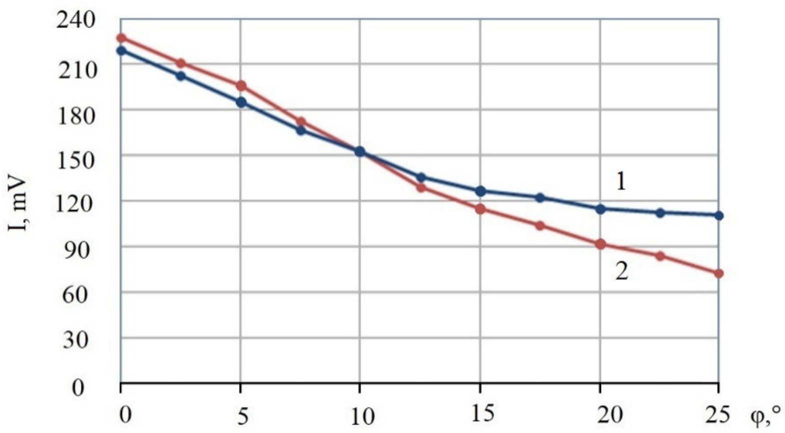


Figure 7 - Changing the intensity of the RM reflection from the angle of incidence of the laser beam. Curve 1 - sample based on an epoxy composition, curve 2 - sample «3M»

The process of obtaining SVM in the form of coverings include: the preparation of the sample surface; applying a polymer powder composition electrostatically to form the reference layer coatings with the prior heat treatment; applying a layer of microspheres; final film formation of powder compositions at optimum temperature-time modes; application of protective transparent layer. It is revealed that a number of developed SVM based on powder coatings and microspheres approach the level of light reflection, and at certain lighting angles exceeds the reference materials of the company "3M". To assess the possibility of practical application of SVM on the basis of PPK, complex studies of physical and chemical properties were conducted, the results of which are shown in the table.

Table - Physical and chemical properties of RM

Characteristics of the	Powdery composition of the			
	PEP-219	PEP-91	P-PE-1130	Pygma P-201
Adgesia, point of achievement	1 per cent	1 per cent	2 per cent	1 per cent
Chemical resistance after contact for 10 minutes: c- with water; c 3% Mr NaCl - with mineral oil	It's a good day. It's a good day. It's a good day.	It's a good day. It's a good day. It's a good day.	It's a good day. It's a good day. It's a good day.	It's a good day. It's a good day. It's a good day.
Lightness is as follows: - 6 hours - 12 hours per hour - 18 hours	No cheating on me. No cheating on me. It doesn't look good. change the color.	No cheating on me. No cheating on me. It doesn't look good. change the color.	No cheating on me. No cheating on me. is insignificant. changing color.	No cheating on me. No cheating on me. It doesn't look good. change the color.
Resistance of coatings to 3% of NaCl solution within 1 hour after ultraviolet treatment within 6 hours	It's a good day.	It's a good day.	It's a good day.	It's a good day.

Thus, the experimental studies made it possible to develop compositions of CBM in the form of coatings and determine the regime parameters of their production that provide the specified performance properties (adhesion, concealment, resistance in atmospheric conditions, resistance in liquid aggressive media, light resistance).

MAIN CONCLUSIONS ON THE WORK

1. Based on the analysis of existing compositions and technologies for obtaining dispersed-filled RM, a scientifically justified choice of initial components for forming films and coatings of various structures was made; their optimal combinations and ratios were determined, as well as optical and physical-chemical characteristics necessary for developing RM with the required technological and operational properties.
2. An experimental setup has been improved to improve the accuracy of measuring the RM reflection indicatrix in a wide range of laser beam incidence angles from 0 to ± 25°.
3. The regularities of the influence of the nature, composition, ratio, combination, dispersion, optical and physical-chemical characteristics of the initial components, as well as the mode parameters of formation on the change of the retroreflective properties of flexible multilayer RM are established.

It is shown that the value of the reflection indicatrix of the developed and reference RM samples decreases as the angle of incidence of the beam increases; moreover, at small angles (up to ±5°), the retroreflective properties of «3M» samples, both with microspheric and angle reflectors, are slightly higher (by 4-6 %), and at angles of incidence from ± 5 to ± 25 are significantly lower than those of materials based on microspheric retroreflectors.

It was found that the maximum effect of retroreflection is observed for RM consisting of a reflective layer in the form of aluminum foil; an intermediate layer of a mixture of aluminum powder of PAP-1 and transparent varnish XB-734 (or a mixture of the powder composition "Metallic silver 9007" and aluminum powder); a binding layer based on a self-adhesive transparent polyester tape; a separating layer in the form of sieves with a dispersion of 48-56 microns, covered with a protective film 90 microns thick, which is applied under excessive pressure at a temperature of 115-120°C; the values of refractive indices of functional layers vary in the range n = 1.47-1.52 and are close to the refractive index of glass microspheres n = 1.54.

4. A method for obtaining a flexible multi-layer thin-film retroreflective material and an installation for its implementation (patent of the Russian Federation for invention No. 2660048 of 04.07.2018), which provides high retroreflective properties, has been developed.

It was found that to ensure high retroreflective properties of materials, the film and the binding layer must have refractive indices close to the refractive index of glass microspheres and differ by no more than 0.5-0.8%. The connection of the protective, sealing and binding layers is carried out by thermal or ultrasonic bonding, and after curing the binder, the formation of the focusing layer on the rear hemisphere of the microspheres is carried out by laying out the heated film. As a carrier tape, a fabric with an anti-adhesive layer is used, on which microspheres are applied electrostatically.

5. Установлены и научно обоснованы закономерности влияния природы, состава, соотношения, дисперсности и оптических характеристик, а также режимных параметров формирования на изменение индикатрисы отражения СВМ в виде покрытий на основе эпоксидных, полиэфирных и эпоксидно-полиэфирных композиций, наполненных стеклянными микросферами.

It is shown that the preliminary heat treatment of powder compositions to form the reference layer RM is in the range температур110-120 °C for 10-15 min; the subsequent deposition of microspheres particle size, 80-100 microns is carried out by an electrostatic spray gun at a speed of 3.5-4.0 m/s at an air pressure of 0.02 kgf/cm^2 on расстоянии150-250 mm from the spray nozzle to the substrate, the voltage on the discharge electrode of the spray gun 60 kV and the short circuit current of 50 μa; the content of the microspheres varies in the range of 15-25 g/m^2; removal of excess microspheres is carried out with an ejector nozzle at a compressed air pressure of 0.025 kgf / cm^2; the final film formation of coatings is carried out at a temperature of 170-185°C for 10-15 minutes, followed by the application of a protective polymer film. The structure was determined and the conditions for maximum interference in multilayer retroreflective coatings based on polymer powder compositions were revealed when the thickness of the intermediate layer and the dispersion of microspheric reflectors changed.

It is established that the achieved level of retroreflective and other basic operational properties of dispersed-filled coatings obtained using powder technology provides high efficiency of the use of RM due to the use of components of domestic production, ease of implementation of the technology with the use of equipment available in operation.

6. Using the results of the research, practical recommendations were developed aimed at obtaining RM of different composition and structure with characteristics that meet the operating conditions.

PUBLICATIONS ON THE TOPIC OF THE DISSERTATION:

Publications in publications recommended by the HAC of the Russian Federation

1. **Vagizov, T. N.** Technologies for obtaining retroreflective coatings with specified properties / T. N. vagizova, E. R. Galimov, Yu. a. Pryakhin / / Bulletin of the Kazan technical University. A. N. Tupolev-KAI.- 2017. - T. 73. - No. 3. - Pp. 49-54.

2. **Vagizov, T. N.** Technologies for obtaining and studying the properties of retroreflective coatings with microspheric reflectors/ T. N. Vagizov/ / Bulletin of the Kazan technical University. A. N. Tupolev-KAI. - 2018.- T. 74. No. 2. - Pp. 29-36.

Publication indexed in the Scopus international database

3. **Vagizov,T.N.** MethodforSynthesisofRetro-ReflectiveCoatingswithSpecified Optical Properties / E.R.Galimov, T.N. Vagizov, A.V.Belyaev // Solid State Phenomena. TransTechPublications. Switzerland: - 2018. - V.284,- pp. 1205-1209.

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