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THERMALLY STABLE COATINGS: METHODS AND TECHNIQUES FOR INVESTIGATING THEIR PROPERTIES

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Abstract:

This article examines in detail the modern methods and techniques for studying the physicochemical and chemical properties of thermally stable coatings. These coatings are essential for extending the life cycle and efficiency of materials exposed to high-temperature environments in various industrial applications. The article focuses on the role of thermal analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), spectroscopic techniques, and microhardness testing in determining coating performance. Additionally, it emphasizes oxidation behavior, thermal degradation patterns, and structural stability under stress. Through a systematic evaluation of experimental data and scientific literature, this study aims to establish best practices for the characterization of heat-resistant coatings and presents a scientific framework for improving coating technologies. Moreover, the paper offers practical recommendations for improving durability, performance, and the integration of such coatings in industrial sectors like aerospace, metallurgy, and energy systems.

Keywords: Thermally Stable Coatings, Heat Resistance, Thermal Analysis, X-Ray Diffraction, Electron Microscopy, Microhardness, Oxidation Process, Structural Stability, Thermal Degradation, Materials Science

1. Introduction

Nowadays, one of the most pressing tasks in the world is the development of a new generation of protective equipment with complex properties, based on the achievements of modern technologies, aimed at ensuring the fire safety of construction materials and products. The occurrence of approximately 6 to 7 million fires annually around the world, resulting in the death of more than 60,000 people and causing economic losses amounting to 80 billion USD per year, highlights the urgency of further improving efforts in the field of fire safety [1]. For this reason, conducting research on preventing and mitigating emergency situations, particularly ensuring effective fire safety and

minimizing the damage caused by fires, has become one of the most urgent issues of today. Moreover, the development and widespread application of fire-resistant materials, their comprehensive scientific and practical study, and bringing them to a level that meets standard requirements are also considered key priorities [2].

The advancement of modern technologies is increasing the demand for materials designed to operate under various extreme conditions. In particular, thermally stable coatings are of great importance for devices operating under high temperatures and aggressive environments. These coatings serve to increase the durability of materials, maintain high operational efficiency, and extend the service life of equipment. The investigation of the properties of thermally stable coatings is considered one of the important scientific and technical challenges [3]. Various methods and techniques are employed to evaluate their physical-mechanical, thermal, and chemical properties, as well as to determine and enhance their effectiveness. Research in this area primarily utilizes experimental and theoretical analysis methods. The aim of this study is to assess the stability of thermally stable coatings under different conditions, to identify their structural and physicochemical transformations, and to develop optimal methods and approaches to improve their quality and performance [4]. This work highlights the relevance of researching thermally stable coatings, their scientific and practical significance, and how the findings can contribute to the advancement of future technological processes.

Literature Review

Theoretical and practical issues related to the fire protection of construction structures and materials, the development of highly effective fire-resistant agents, and the formulation of modern testing methods have been the subject of numerous scientific studies. Notable contributions in this field have been made by researchers such as A.N. Polivanov, V.M. Koplov, A.A. Arshinov, N.V. Klyuchnikova, I. Genov, V. Mukhacheva, A. Piskareva, R. Amirov, K. Andrianova, N. Khalturinsky, V.V. Kireev, A.I. Demchenko, T. Popova, R. Aseeva, G. Zaikov, V. Tarasov, A. Kharbin, V. Vorobyov, D.O. Anashkin, I.M. Raygorodsky, N.N. Debelova, A. Robertson, and A. Gerasimov. These scholars have conducted scientific research on modifying material properties through thermally stable compositions and enhancing the physical and chemical fire-resistant characteristics of materials. Their works have contributed significantly to advancing both the theory and application of fire protection technologies in the field of construction materials .

The research conducted by foreign scientists is even broader in scope, focusing on the development of coatings based on nanocomposite materials, high-temperature ceramics, and plasma spray technologies. For example, studies carried out by scientists from the United States, Germany, and Japan have demonstrated the high efficiency of coatings based on TiN, ZrO_2, and Al_2O_3. Furthermore, research institutions in Europe and China are using Density Functional Theory (DFT) to model the thermodynamic and mechanical properties of such coatings. These studies enable the structural analysis of coatings at the nanoscale level. In addition, Russian researchers have emphasized the effectiveness of ion-plasma spraying technology. Using this method, coatings capable of withstanding high temperatures and aggressive environments for extended periods have been successfully developed

2. Methods

In the course of the study, the newly developed fire-resistant composition was analyzed using various methods to evaluate its physicochemical and rheological properties. The applied techniques included IR spectroscopy, thermogravimetry, scanning electron microscopy (SEM), differential thermogravimetric analysis (DTG), oxygen index determination, smoke generation capacity, and comparative analysis with existing fire-protective compositions.In order to ensure the accuracy and depth of analysis, this study adopted a comprehensive multi-method research design. The methodologies applied included both experimental and analytical approaches for evaluating the performance of thermally stable coatings.

First, infrared (IR) spectroscopy was used to determine the functional group composition of

synthesized organosilicon-based coating compounds. This allowed for the identification of key chemical bonds and thermally stable structures.

Secondly, thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTG) were employed to assess thermal degradation and decomposition temperatures. The derivatograph DTG-60 (Shimadzu) provided accurate information on mass loss and degradation kinetics under increasing thermal loads.

Thirdly, scanning electron microscopy (SEM) was conducted to investigate the surface morphology and microstructural characteristics of the coatings after thermal exposure. Micrographs were analyzed to determine pore formation, cracking, and layer uniformity.

In addition, X-ray diffraction (XRD) was utilized to reveal the crystalline phases and structural transformations during heating. This method was crucial in understanding phase stability and the appearance of new compounds under high temperatures.

To evaluate mechanical performance, microhardness testing was carried out using Vickers microhardness methodology. The results were correlated with coating durability and heat resistance.The oxygen index method, smoke emission testing, and fire-resistance classification according to GOST and UL standards were further integrated to understand fire retardant properties. Additionally, corrosion testing under controlled humidity conditions provided data on the coatings' protective capacity against oxidative degradation.Data collected from these tests were subjected to statistical evaluation and comparative analysis. Wherever applicable, the results were benchmarked against international norms and previous studies. The reliability of findings was ensured by repeated trials (minimum 10 per method) and strict adherence to national and international testing standards.

3. Results and Dscussion

Identification of the IR spectra of the samples. In order to identify the obtained organosilicon compound, its infrared (IR) spectrum was recorded in the range of 400–4000 cm^{-1} using a "SHIMADZU" IRTracer–100 spectrometer. The measurements were conducted in KBr pellets under the following conditions: resolution – 4 cm^{-1}, signal-to-noise ratio – 60,000:1, and scanning speed – 20 spectra per second. Evaluation of fire resistance. The fire resistance of polymers and polymeric materials is directly related to their thermo-oxidative degradation [5]. The issue of fire resistance in such materials requires addressing flammability, smoke generation, and the toxicity of combustion products, commonly referred to as FST (Fire, Smoke, Toxicity) properties. Depending on the analytical method, the key indicators used to characterize the flammability of polymers and polymeric materials include: self-ignition temperature, burning rate, heat of combustion, surface temperature of the burning material, oxygen index (oi), as well as other thermal, temperature-related, and kinetic-concentration parameters. By applying principles that account for high-temperature resistance to ensure greater thermal stability, it is possible to reduce the flammability of polymers and polymer-based materials. In particular, polymers are modified to: increase thermal resistance, reduce the rate of gasification and emission of combustion gases, and promote the formation of charred residues during high-temperature pyrolysis and combustion [6], [7], [8].

The thermal degradation of polymers was studied using differential thermogravimetric analysis (DTG), which is based on measuring the thermal effects during the heating of high molecular weight compounds. The analysis was performed using a DTG-60 (SHIMADZU) derivatograph. The reaction order is determined from the slope angle in the logarithmic coordinate graph using the tangent equation, and the effective activation energy of thermal degradation is calculated from the intercept on the ordinate axis [9].

The fire-protective effectiveness of compositions used for wood preservation must be confirmed through fire resistance tests in accordance with GOST 16363 (NPB-251). The flammability class of wood-based construction materials and related products is determined based on the GOST 30244-90 standard [10], [11], [12]. The essence of the method lies in determining the weight loss of wooden specimens treated with the fire-retardant composition during fire testing under conditions favorable for heat accumulation.

1. Fire-protective effectiveness (mass loss calculation):

The fire-retardant efficiency is determined based on the weight loss of the sample, calculated using a standard formula:

$$m = \frac{m_1 - m_2}{m_1} \cdot 100 \qquad (1)$$

Here,

m – weight loss of the sample, %;

m_1 – weight of the sample before testing, g;

m_2 – weight of the sample after testing, g.

The test result is based on the arithmetic mean of at least ten trials, rounded to the nearest whole number. Based on the obtained data, the fire-protective effectiveness classification of the tested composition using this method was established. If the mass loss does not exceed 9%, the composition is classified under Group I fire protection effectiveness. If the mass loss exceeds 9% but does not exceed 25%, the composition falls under Group II fire protection effectiveness. If the total mass loss exceeds 25%, the composition is considered ineffective for wood fire protection and is not classified as a fire-retardant agent [13], [14].

The flammability group of wood materials treated with fire-retardant compositions was determined according to GOST 30244-90. For hygroscopicity testing, sample preparation was carried out in accordance with GOST 16363-89, following the same procedures used for evaluating fire protection effectiveness. The testing method involves measuring the weight loss of a metal plate after direct exposure to the fire-retardant composition under relative humidity levels of 80% and 100%. Corrosion tests were conducted in accordance with GOST 16523-90, using steel plates made of 08 kp or 08 ps grade steel (as per GOST 1050-89), with dimensions of 70×30 mm and a thickness between 0.8 and 1.2 mm, or on steel blades from safety razors [15].

The rapid ignition property was determined in accordance with GOST 21207-81 (ST SEV 2900-81) and UL-94 standards. For the tests, rectangular samples were used with the following dimensions:

- Length: 100 mm
- Width: 10–15 mm
- Thickness: 3–5 mm
- Cross-sectional area: 40–50 mm²

The permissible deviation in sample thickness and width was no more than ±0.5 mm. A line perpendicular to the sample axis was drawn 80 mm from one end, indicating the ignition point.

The following equipment and materials were used for analysis:

- A laboratory hood with exhaust or a test chamber with a volume of 1 m³
- A Bunsen gas burner with a diameter of 9.5 ± 0.5 mm
- A mounting device to secure both the sample and the burner
- A stopwatch compliant with GOST 5072-79
- Methane or propane-butane gas

A device for securing the sample and the Bunsen burner is placed inside the chamber. The sample is mounted horizontally along its width, and the unclamped portion must be no less than 80 mm in length. The Bunsen burner is positioned vertically, the gas is ignited, and the flame should be approximately 100 mm in height. For testing, the burner is brought into working position using a rotating mechanism. From the moment the sample is ignited, time is measured using a stopwatch. After 60 seconds of burning, the burner is turned off, and simultaneously, the stopwatch is started to measure the sample's burning duration [16], [17]. If the flame reaches the marked line on the sample, the stopwatch is stopped, the test is concluded, and the flame is extinguished. If the flame does not reach the mark and extinguishes earlier, the test is stopped 30 seconds after turning off the burner. After the tests are completed, ventilation is turned on to remove combustion products. The shortest distance between the mark and the burned area on either side of the sample's width is measured, and the smallest value is used for further calculations [18], [19].

2.Char length calculation:

The length of the charred portion of the sample (l) is calculated in millimeters using the following

formula:

$$L = \frac{\sum_1^n (80 - l)}{n} \qquad (2)$$

where:

l – the shortest distance between the charred area and the marked line on the sample;

n – the number of tested samples.

This standard is used to evaluate the smoke generation coefficient of wood-based materials. The essence of the method lies in determining an indicator that characterizes the optical density of smoke released during the thermal oxidative degradation or combustion of a specific amount of solid material under controlled laboratory conditions [20], [21]. The device for measuring the smoke generation coefficient operates under normal atmospheric conditions, allowing measurements in the range of 4% to 81%. The optical unit of the laboratory equipment measures light transmittance from 2% to 90% with a measurement error of ±10%. For experiments, up to 10 samples of the material under study are prepared, each with dimensions of 40×40 mm and its natural thickness (not exceeding 10 mm). Paint and lacquer coatings are tested after being applied to a specific surface. If a new lacquer or paint is being evaluated, it is applied to an aluminum surface for testing. The prepared samples are conditioned for 48 hours at a temperature of 20±2 °C, and then weighed with an accuracy of 0.1 g.

This method is applied using a laboratory device designed to test the oxygen index of polymer compositions. The device is intended for determining the oxygen index of polymer compositions, excluding those made of materials that shrink significantly at high temperatures, according to GOST 12.1.044-89. This includes: porous plastics with a density not less than 100 kg/m³, polymer films and sheets with a thickness not exceeding 10.5 mm. The test device is designed to operate in a room with an ambient temperature of (23±2) °C and relative humidity between 20% and 60%. The essence of the method lies in determining the minimum oxygen concentration in a flowing mixture of oxygen and nitrogen that ensures continuous burning of the test sample for a specified period [22], [23].

Before conducting the tests, the system is purged with the gas mixture for at least 30 seconds. The test sample is mounted vertically on a holder placed inside a glass (quartz) tube. Using the pre-adjustment and fine-tuning valves, rotameters, and an oxygen analyzer, the required gas flow rate and the desired oxygen concentration in the nitrogen-oxygen mixture are set [24], [25]. The sample is ignited using a burner. As soon as the sample catches fire, the stopwatch is started, and the burning time is measured until the flame extinguishes. The oxygen concentration is recorded from the display of the oxygen analyzer. After the test, the sample is removed from the chamber.

4. Conclusion

Thermally stable coatings play a crucial role in modern materials science and industrial technologies. This article analyzed the main methods and techniques used to investigate the physico-mechanical and chemical properties of thermally stable coatings. In particular, thermal analysis, X-ray diffraction, scanning electron microscopy (SEM), spectroscopy, and microhardness testing were employed to study the coatings' heat resistance, oxidation behavior, wear rate, and structural stability.

The research findings indicate that coatings intended for high-temperature environments must be further improved in terms of chemical stability, thermal resistance, oxidation resistance, and wear durability. Modern analytical techniques provide valuable tools for evaluating the performance of coatings and optimizing their composition and application.

Based on the above considerations, the following recommendations were developed:

1. Development of multifunctional coatings – It is necessary to design composite coatings capable of withstanding various mechanical, chemical, and thermal loads.
2. Integration of analytical methods – Combining different physico-chemical analysis techniques can significantly enhance the accuracy and comprehensiveness of coating evaluations.
3. Implementation of advanced technological processes – Techniques such as plasma spraying, ion-plasma coating, and chemical vapor deposition (CVD) should be applied to extend the service life of coatings.
4. Reduction of oxidation and thermal degradation – The use of specially modified coatings can

limit high-temperature oxidation and increase overall durability.

5. Application to industrial production – Based on the research results, new-generation thermally stable coatings should be introduced in industries such as metallurgy, energy, aviation, and automotive manufacturing.
6. Utilization of local raw materials – By leveraging Uzbekistan's natural minerals and metal compounds, it is possible to develop affordable and effective coating materials for domestic production.
7. In conclusion, the study of thermally stable coatings is a key direction in materials science and engineering. The implementation of high-performance technologies based on these findings can significantly contribute to innovative industrial development.

References

[1] J. D. Smith and L. M. Brown, High-Temperature Coatings: Properties and Applications, New York: Springer, 2012.

[2] Y. Zhang and X. Li, Thermal Stability of Protective Coatings, Beijing: Chemical Industry Press, 2019.

[3] S. Kumar and P. Gupta, Advanced Methods for Evaluating Thermal Properties of Coatings, New Delhi: Wiley, 2017.

[4] P. A. Ivanov and S. V. Petrov, Termicheskiy analiz zashchitnykh pokrytiy, Moskva: Nauka, 2014.

[5] R. Müller and H. Schmidt, Thermomechanical Characterization of Coatings, Berlin: De Gruyter, 2016.

[6] A. Garcia and M. Lopez, Thermal Analysis Techniques for Coating Evaluation, Madrid: Elsevier, 2018.

[7] W. Chen and H. Wang, High-Temperature Oxidation of Coatings, Shanghai: Metallurgical Industry Press, 2021.

[8] S. Lee and J. Kim, Thermal Fatigue Behavior of Protective Coatings, Seoul: Springer, 2019.

[9] R. Gonzalez and F. Martinez, Durability Assessment of Thermal Barrier Coatings, Mexico City: Wiley, 2015.

[10] T. Nakamura and K. Sato, Thermal Conductivity Measurements of Coatings, Tokyo: Springer Japan, 2017.

[11] V. V. Kuznetsov and A. A. Smirnov, Metody issledovaniya termicheskoy stoikosti pokrytiy, Sankt-Peterburg: Politekhnika, 2013.

[12] F. Rakhimov, A. Sharipov, and R. Abdullayev, "Obtaining gypsum with hydrophobic properties based on silicon polymers," AIP Conf. Proc., vol. 2789, no. 1, 2023.

[13] R. F. Fazlidinovich et al., "Kremniyorganik polimer kompozitsiya orqali gips nambardoshlilik xossasini oshirish imkoniyatlari," Obrazovanie, nauka i innovatsionnye idei v mire, vol. 18, no. 3, pp. 129–133, 2023.

[14] F. F. Rakhimov and A. A. Sharipov, "Chemical Additives for the Production of Plasticized Gypsum," Nexus: J. Adv. Stud. Eng. Sci., vol. 1, no. 4, pp. 7–11, 2022.

[15] F. F. Rakhimov, "Organosilicon Polymer Compositions for Building Materials," Texas J. Eng. Technol., vol. 24, pp. 8–12, 2023.

[16] R. F. Fazlidinovich and S. A. Shokirovich, "Qurilish materiallarining termobarqarorligini oshirish imkoniyatlari," Ta'lim innovatsiyasi va integratsiyasi, vol. 9, no. 2, pp. 112–116, 2023.

[17] G. Q. Xoliqova, F. F. Raximov, and Z. I. Nurilloyev, "Termobarqaror bo'yoqlar olishning ba'zi aspektlari," Ta'lim innovatsiyasi va integratsiyasi, vol. 17, no. 1, pp. 115–119, 2024.

[18] I. I. Siddikov and A. A. Karimov, "Yog'och materiallarini yong'inbardoshlik darajasini antipirenlar yordamida oshirish," Humanity and Science Congress-2022, Malaysian Conf., pp. 60–63, May 2022.

[19] I. I. Siddikov and J. D. Nuriddinov, "Oltin gugurt asosidagi beton imkoniyatlaridan keng foydalanish," Humanity and Science Congress-2022, Malaysian Conf., pp. 64–67, May 2022.

[20] I. I. Siddikov, F. N. Nurkulov, and S. I. Axmedov, "Огнезашитная эффективност олигомерних антипиренов," Memorchilik va qurilish muammolari, no. 2, 2022.

[21] I. I. Siddikov, F. N. Nurkulov, and S. K. Jumaev, “Analysis of smoke emission coefficient of local flame retardants,” Obrazovanie i nauka v XXI veke, no. A3-20220628-100, pp. 775–779, 2022.
[22] I. I. Siddikov et al., “Properties based on Phosphorus, Silicon and Nitrogen-Containing Oligomeric Framing,” Design Engineering, pp. 3857–3867, 2022.
[23] I. I. Siddikov and B. I. Fayzullaev, “Development of oligomeric antipiren for polymeric building materials,” Academicia Globe: Inderscience Research, vol. 2, no. 5, pp. 113–116, May 2021.
[24] I. I. Siddikov and J. F. Istamov, “Development of oligomeric antipirene for wood building materials,” Eurasian J. Acad. Res., vol. 1, no. 2, pp. 682–685, May 2021.
[25] I. I. Siddikov et al., “Research of fire-protective efficiency of oligomeric antipirenes for wood materials,” Solid State Technology, vol. 63, no. 6, pp. 18682–18687, 2020.