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**Sereda Dmytro**, Candidate of Technical Sciences, Associate Professor, Department of industrial engineering

**Серєда Д.Б.**, кандидат технічних наук, доцент кафедри галузевого машинобудування

ORCID: 0000-0003-4353-1365

e-mail: seredabp@ukr.net

**Udod Andrey**, postgraduate student, Department of automobiles and transport and logistics systems

**Удод А.М.**, здобувач третього (доктора філософії) рівня вищої освіти, кафедра автомобілів та транспортно-логістичних систем

Dnipro State Technical University, Kamianske

Дніпровський державний технічний університет, м. Кам'янське

## MATHEMATICAL MODELING OF THE DEVELOPMENT OF RATIONAL FUNCTIONALLY ACTIVE CHARGES IN CHROME PLATING OF CARBON-CARBON MATERIALS

### МАТЕМАТИЧНЕ МОДЕЛЮВАННЯ РОЗРОБКИ РАЦІОНАЛЬНИХ ФУНКЦІОНАЛЬНО АКТИВНИХ ШИХТ ПРИ ХРОМУВАННІ ВУГЛЕЦЬ-ВУГЛЕЦЕВИХ МАТЕРІАЛІВ

*Research is devoted to the development of high-performance protective coatings for carbon-carbon composite materials (CCCM) designed to operate under extreme thermal loads. An innovative approach to the formation of coatings by using functionally active charges (FAC) in non-stationary temperature conditions is proposed, which ensures the formation of a two-layer structure with increased heat resistance. The effect of alloying with titanium, chromium, aluminum, and silicon on the structural and phase characteristics of coatings was experimentally studied using X-ray diffraction, metallography, and thermodynamic analysis. It was found that the optimal composition of the FAS with concentrations of titanium of 10—15 wt%, chromium of 20—25 wt%, aluminum of 15—20 wt%, and silicon of 5—10 wt% promotes the formation of layer of titanium carbide (TiC) with layer of intermetallic phases  $Al_2Cr_3$ ,  $CrAl_2$ , and  $TiAl$ . These coatings demonstrate an activation energy of diffusion in  $\gamma$ -iron of 250.8 kJ/mol. Compared to isothermal methods, the coatings formed using FAC have lower porosity and higher oxidation resistance due to the formation of protective oxides  $SiO_2$ ,  $TiO_2$ ,  $Cr_2O_3$  and  $Al_2O_3$ , which reduce the penetration of oxygen into the CCCM matrix. The factor experiment and regression analysis allowed us to build three-dimensional models of the dependence of heat resistance on the composition of the charge, confirming the optimality of the multistage mechanism of coating formation, where the primary  $CrAl$  layer is supplemented by high-temperature phases of  $TiAl$  and  $CrSi_2$ . The resulting coatings increase the heat resistance of CCCM by 40% compared to traditional chemical-thermal methods, making them promising for use in the aerospace and energy industries, where high resistance to aggressive environments is required.*

**Keywords:** protective coatings, carbon-carbon composite materials, heat resistance, synthesis, optimization, chemical and thermal treatment.

*Наше наукове дослідження зосереджено на розробці ефективних захисних покриттів для вуглець-вуглецевих композиційних матеріалів (ВВКМ), що функціонують в умовах екстремального теплового навантаження. У роботі застосовано функціонально активні шихти (ФАС), які формуються за нестационарних температурних режимів, з метою отримання покриттів із підвищеними експлуатаційними характеристиками. Основна мета полягає в розробці та оптимізації складу порошкових композицій, легованих титаном, що сприяють підви-*

ценню жаростійкості функціональних поверхонь ВВКМ. У межах дослідження було здійснено комплексний аналіз існуючих технологій нанесення захисних покриттів, зокрема хіміко-термічних методів та методів насичення з рідкої фази. Розглянуто їхню ефективність з урахуванням специфіки взаємодії з вуглецевою матрицею та змін у механічних властивостях матеріалу. Особливу увагу приділено вивченню альтернативного методу — насичення поверхні в твердофазному стані в присутності активного газового середовища, що реалізується за допомогою ФАШ, сформованих за умов нестационарного термічного впливу. Значний акцент зроблено на вивченні хіміко-фізичних процесів, зокрема утворення карбідних фаз, які відіграють ключову роль у забезпеченні стійкості покриттів у агресивному високотемпературному середовищі. Експериментальна частина включає постановку факторного експерименту для визначення оптимального складу порошкових сумішей, що забезпечують максимально можливу термостійкість захисного шару. У якості незалежних змінних досліджено концентрації хрому, кремнію, титану та алюмінію, враховуючи їхній вплив на структурно-фазовий стан та фізико-механічні параметри покриттів. Побудовано рівняння регресії для кількісної оцінки залежності жаростійкості від параметрів автоініційованої термічної обробки та складу легуючих компонентів. Результати аналізу представлені у вигляді тривимірних графічних моделей, що ілюструють оптимізацію складу порошкових ФАШ у системах Cr–Al–Ti. За результатами структурно-фазового аналізу встановлено, що при легуванні титаном захисне покриття формується як двозонна система: внутрішній шар представлений фазою карбіду титану (TiC), тоді як зовнішній шар утворюється із сполук типу  $Al_2Cr_3$ ,  $CrAl_2$  та  $TiAl$ , хімічний склад яких визначається варіацією компонентного складу ФАШ.

**Ключові слова:** захисні покриття, вуглець-вуглецеві композиційні матеріали, жаростійкість, синтез, оптимізація, хіміко-термічна обробка.

### Formulation of the problem

This research is focused on the comprehensive optimization of technological regimes for the The increasing operational demands placed on modern aerospace and energy systems have intensified the need for advanced thermal protection solutions, particularly for structural elements manufactured from carbon-carbon composite materials (CCCM). These materials, despite their exceptional thermal and mechanical performance, exhibit inherent vulnerability when exposed to prolonged high-temperature oxidizing environments and high-velocity gas flows. Consequently, the enhancement of their functional stability through the development of protective coatings has emerged as a critical area of scientific inquiry

This research addresses the scientific and technological challenge of optimizing the formation regimes of protective coatings on CCCM components. The investigation emphasizes the physicochemical and structural evolution of the coating systems under extreme thermal conditions. In this context, particular focus is given to elucidating the mechanisms of phase transformations occurring within the coating matrix, which govern its resistance to thermal degradation, oxidation, and erosive damage.

A central objective of the research is to provide a scientific rationale for the composition of a functionally active charge (FAC) tailored for the deposition of titanium-alloyed protective coatings. These coatings are engineered to withstand aggressive thermo-gas-dynamic conditions, characterized by intense jet flow exposure and cyclic thermal loads. The research involves a systematic comparison of competing coating technologies—namely, chemical-thermal diffusion methods and liquid-phase saturation processes—with the aim of assessing their respective impacts on the microstructure, adhesion, and mechanical integrity of the treated composite substrates.

Special attention is devoted to the selection and synergistic balance of alloying elements within the protective layer. Elements such as titanium, silicon, aluminum, and chromium are strategically integrated to promote the formation of stable, adherent oxide films that function as diffusion barriers. These films play a pivotal role in suppressing high-temperature oxidation kinetics and maintaining the structural cohesion of the composite during service. The optimization of these compositional parameters is essential for extending the operational lifespan of CCCM-based components under severe service conditions.

### **Analysis of recent research and publications**

The operational implementation of CCCM in advanced engineering applications continues to be constrained by a series of critical challenges, the most prominent of which include their inherent susceptibility to high-temperature oxidation, erosion, and thermally induced degradation under the influence of reactive gas flows. These failure mechanisms significantly compromise the reliability and longevity of CCCM components in extreme thermal environments. Consequently, a central research priority has emerged: the development and application of thermochemically stable protective coatings capable of mitigating these deleterious effects and extending the operational service life of CCCM-based structures.

Among the materials demonstrating high potential for use as protective coatings are refractory compounds such as carbides, borides, nitrides, and silicides. These ceramic-based materials are distinguished by their superior resistance to oxidative attack, exceptional hardness, and outstanding wear performance under both thermal and mechanical loads. Their application as surface barriers enables effective shielding of CCCM substrates from aggressive high-temperature environments, thereby preserving structural integrity and functionality under extreme operating conditions.

Carbon-carbon composites, composed of carbon or graphite matrices reinforced with high-modulus carbon or graphite fibers, exhibit a unique combination of physicochemical properties that render them indispensable in advanced aerospace and energy systems. These include low specific density, high tensile and compressive strength at elevated temperatures, and remarkable resistance to thermal shock and fatigue phenomena. The integration of such materials has found particularly extensive use in the aerospace and defense industries—most notably in components of solid-propellant rocket motors, where combustion chamber environments routinely exceed 2500 °C. Under these conditions, material systems are subjected to intense abrasive action from high-velocity particle-laden exhaust streams, necessitating the implementation of robust ablative and erosion-resistant protective strategies [2].

Even more stringent thermal requirements are encountered during the atmospheric re-entry phase of spacecraft, wherein leading-edge components can be exposed to temperatures in excess of 5000 °C due to aerodynamic heating. In such scenarios, only materials exhibiting the highest levels of thermal and oxidative stability are viable. The significant technological progress in the field of CCCM over the past decades is inextricably linked to advancements in the development of carbon fibers and other thermally stable fibrous reinforcements. The microstructural characteristics and thermal stability of these reinforcement phases play a decisive role in dictating the overall performance and durability of the composite system. As a result, the careful selection and engineering of fiber types, orientations, and volume fractions remain a cornerstone of ongoing innovation in CCCM technologies.

Within the framework of the present research, the primary methodological focus is centered on the identification and optimization of a FAC formulation, specifically designed to enable the deposition of lightweight chromium-based coatings onto CCCM substrates under non-stationary thermal loading conditions. This process aims to address the dual challenge of ensuring thermal protection and minimizing the mass contribution of the coating layer, which is of particular importance in aerospace and high-speed flight applications [3].

A variety of technological approaches have been investigated for the formation of protective coatings, each characterized by its own set of operational benefits and inherent limitations. One of the more complex processes involves the introduction of silicon into the CCCM surface layer through liquid-phase saturation. This method often initiates chemical interactions with the carbon matrix, resulting in heterogeneous formation of carbide phases and consequent alterations in the mechanical behavior of the composite material. Despite these challenges, several techniques—such as chemical-thermal processing and saturation from a liquid medium—have been successfully implemented for the modification of carbon-based substrates [4,5].

Among the more advanced and promising solutions is the diffusion-based saturation from a solid phase in the presence of an active gaseous environment, using a functionally active charge (FAC) under non-stationary thermal conditions. This chemical-thermal method has demonstrated the ability to generate protective coatings of high structural quality, reduce process duration, and achieve elevated treatment temperatures, which are directly influenced by the composition of the FAC. By fine-

tuning the quantitative and qualitative content of alloying components, it becomes possible to tailor the resulting coating compositions to satisfy specific operational demands.

Chromoaluminizing technology has garnered significant interest for its role in enhancing the thermal durability of aerospace structural components. The process—whether through concurrent or staged saturation with chromium and aluminum—is primarily aimed at increasing wear resistance, heat resistance, and chemical stability of metal parts operating under extreme conditions [6]. Techniques of chromium aluminizing encompass a wide range of process modalities, including solid-state, vapor-phase, gaseous, and molten-phase treatments, with options for simultaneous or sequential application. These methodologies allow for considerable flexibility in engineering coatings with specific functional characteristics adapted for high-demand sectors, particularly in aviation and astronautics.

The alloying of chromium-based coatings with additional elements such as titanium, silicon, and boron is considered a highly promising direction for improving protective performance. Incorporating these elements leads to the formation of complex oxide phases—such as titanium and silicon oxides—which contribute to increased surface hardness, improved scale resistance, and enhanced corrosion protection, thereby significantly broadening the functional capabilities of the resulting coatings [7,8].

### **Formation of the purpose of the research**

The main objective of this Research is to determine and justify the optimal technological parameters for creating protective coatings on carbon-carbon composite materials, with a focus on examining their phase composition and improving their thermal stability under extreme operational conditions. The key scientific task is the development of a functionally active charge designed for the synthesis of alloyed titanium-based coatings that exhibit high resistance to oxidation, erosive wear, and aggressive gas-phase environments.

To this end, a comparative analysis of several coating techniques—including chemical-thermal treatment and methods based on saturation from the liquid phase—was conducted to assess their respective efficiencies and influence on the resultant mechanical properties of the coated materials. Special emphasis is placed on the precise determination of the optimal chemical composition of the protective layer, incorporating the influence of alloying elements such as silicon, titanium, aluminum, and chromium. These components are essential for the formation of stable oxide films (e.g.,  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ) that serve as diffusion barriers and contribute to the preservation of the mechanical integrity of the underlying composite substrate.

Experimental investigations are planned to determine the heat treatment regimes that most effectively promote coating formation and to establish the interrelationship between FAC composition and the mechanical performance metrics of the protective layers. The research particularly emphasizes the study of physicochemical interactions and reaction mechanisms occurring during the coating process, including the nucleation and growth of oxide phases and accompanying structural transformations within the composite material matrix.

### **Presenting main material**

The obtained experimental data and their analytical interpretation constituted a critical component of the research process. Through comprehensive investigation of physicochemical reactions occurring under non-isothermal (non-stationary) thermal regimes, in conjunction with metallographic characterization, a conceptual model for the synthesis of protective coatings was formulated. The sequence of coating formation under thermal autoignition conditions can be systematically segmented into five principal stages: (1) initial inert heating of the FAC to the autoignition threshold; (2) spontaneous thermal ignition of the reactive medium; (3) heating of the carbon-carbon composite components; (4) isothermal exposure or holding period; and (5) gradual cooling to ambient temperature.

The thermal stability of the obtained coatings was assessed via prolonged exposure in an electric resistance furnace, maintained at 1000 °C over a continuous duration of 25 hours. The primary indicator of thermal resistance was defined as the mass variation of the coated specimen following high-temperature treatment.

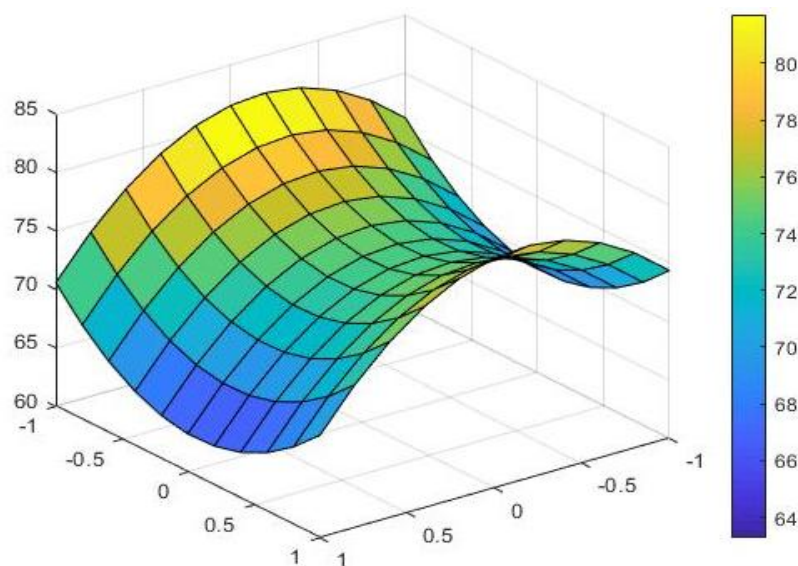
In the experimental design, the concentration levels of chromium, silicon, titanium, and aluminum in the FAC mixture were selected as independent variables. CCCM served as the substrate for all experiments, while iodine ( $\text{I}_2$ ) and ammonium fluoride ( $\text{NH}_4\text{F}$ ) were systematically introduced as chemical activators to stimulate mass transport phenomena. It was determined that the addition of gas-

phase transport agents in quantities exceeding 5 % resulted in pronounced surface degradation and topological distortion of the composite, whereas concentrations below 1% were insufficient to initiate complete activation of the gas-phase transport reactions.

To balance the overall FAC composition to 100 %, aluminum oxide ( $\text{Al}_2\text{O}_3$ ) was employed as an inert filler. In order to quantify the influence of experimental parameters on coating performance, a regression analysis was carried out. This enabled the derivation of mathematical models describing the dependence of wear resistance on both the thermal autoignition regime and the specific content of alloying constituents. The established regression equations are presented below:

$$Y = 123,7 + 0,6X_1 - 2,3X_2 - 1,8X_3 - 5,2X_1^2 + 8X_2^2 + 4X_3^2 - 0,375X_1X_2 + 2,125X_1X_3 - 1,125X_2X_3.$$

The reliability and predictive power of the constructed mathematical models were evaluated and confirmed through statistical adequacy analysis. The models demonstrated sufficient accuracy in forecasting the behavior of the response functions across the entire spectrum of investigated factor levels, thereby validating their suitability for practical application within the defined experimental boundaries. To determine the optimal combination of parameters ensuring maximum thermal resistance of the protective coatings, both the thermal treatment regime and the compositional variables of the functionally active charge were systematically analyzed. For the purpose of visualizing the influence of these parameters, three-dimensional surface plots were generated (Fig. 1), which clearly illustrate the multifactorial relationships and their effects on the heat resistance characteristics.



*Fig. 1.* Influence of titanium and aluminum concentration in FAC mixtures on the oxidation resistance of protective coatings

During the thermal testing phase, samples were periodically weighed at five-hour intervals using high-precision analytical balances VLR-200 with a resolution of  $10^{-4}$  grams. The total duration of the exposure experiment was 25 hours. This allowed for the assessment of oxidation kinetics and mass loss associated with thermal degradation.

The gas-phase transport method, employing functionally active mixtures, enables the deposition of protective layers on CCCM substrates while simultaneously enhancing their physical and mechanical characteristics. A key advantage of this approach lies in its low energy demands and reduced process duration, which makes it highly efficient compared to conventional methods.

The temperature profile of the reaction mixture during the FAC-induced process is governed by two main factors: the preliminary heating temperature and the enthalpy released during the reduction reaction. It is important to emphasize that the ultimate temperature attained during the process is a function not only of the heat released during the redox interaction but also of the specific thermal interval in which the reaction proceeds.

Aluminum acts as the principal alloying element in all high-temperature oxidation-resistant coatings. Its function is to facilitate the formation of a dense and stable protective oxide layer consisting of  $\alpha\text{-Al}_2\text{O}_3$ . In diffusion-type protective systems, the aluminum content typically ranges from 15 to 25 %, which is sufficient to generate a continuous alumina scale under oxidative conditions. Chromium serves as the second most critical component, contributing to the long-term stability and regeneration of the  $\alpha\text{-Al}_2\text{O}_3$  layer. For chromium, the content in advanced thermal barrier coatings generally lies within the range of 7 to 20 %.

The protective coatings synthesized under FAC regimes on CCCM substrates alloyed with titanium exhibit a diffusion-controlled growth mechanism and are characterized by uniformity in thickness. Notably, an increase in processing temperature leads to a proportional increase in the overall coating thickness (Fig. 2), indicating the thermally activated nature of the diffusion process.

Results obtained through X-ray phase analysis and metallographic investigation of protective layers formed on high-temperature carbon–carbon composite materials (HCCM) confirm the presence of a two-zone structure throughout the entire temperature–time interval of processing. The internal region of the coating predominantly consists of titanium carbide (TiC), while the composition of the external zone is determined by the alloying system incorporated into the functional charge. In the case of titanium alloying, the external phase constituents include complex intermetallics such as  $\text{Al}_2\text{Cr}_3$ ,  $\text{CrAl}_2$ , and  $\text{TiAl}$  (Fig. 3).

Upon simultaneous saturation with chromium, aluminum, and titanium, the formation of solid substitutional solutions occurs within the surface layer. The activation energy ( $Q$ ) for atomic diffusion in  $\gamma$ -iron under these conditions exceeds 250.8 kJ/g-atom. Consequently, the application of a non-isothermal (non-stationary) heating regime not only facilitates rapid attainment of elevated temperatures but also enhances the concentration gradient of diffusing alloying species at the material's surface.

In contrast, coatings fabricated under conventional isothermal processing conditions exhibit a more porous morphology and typically include the  $\text{FeAl}$  intermetallic phase. The porosity allows oxygen ingress, which in turn compromises the oxidation resistance by permitting oxygen access to the substrate. Protective layers synthesized under FAC-assisted thermal regimes demonstrate superior structural integrity and oxidation resistance. This improvement arises from the increased incorporation of chromium, aluminum, silicon, and titanium, which promote the formation of thermodynamically stable oxide scales, including  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_3$ , and  $\alpha\text{-Al}_2\text{O}_3$ .

An important characteristic of the thermal barrier systems produced via FAC is the sequential nature of protective layer formation. Initially, a  $\text{Cr-Al}$ -based layer is formed, which subsequently undergoes in-situ alloying with titanium, chromium, and silicon. This sequence leads to the formation of thermally stable intermetallic compounds such as  $\text{TiAl}$  and  $\text{CrSi}_2$ , thereby significantly enhancing the coating's capability to withstand extreme thermal and chemical stressors.

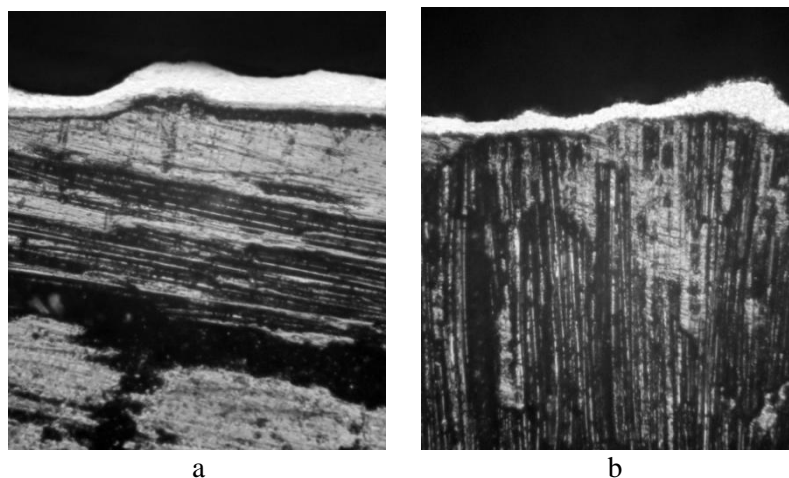


Fig. 2. Surface morphology of titanium-based protective coatings after various exposure durations: a) 20 minutes, b) 60 minutes,  $\times 100$

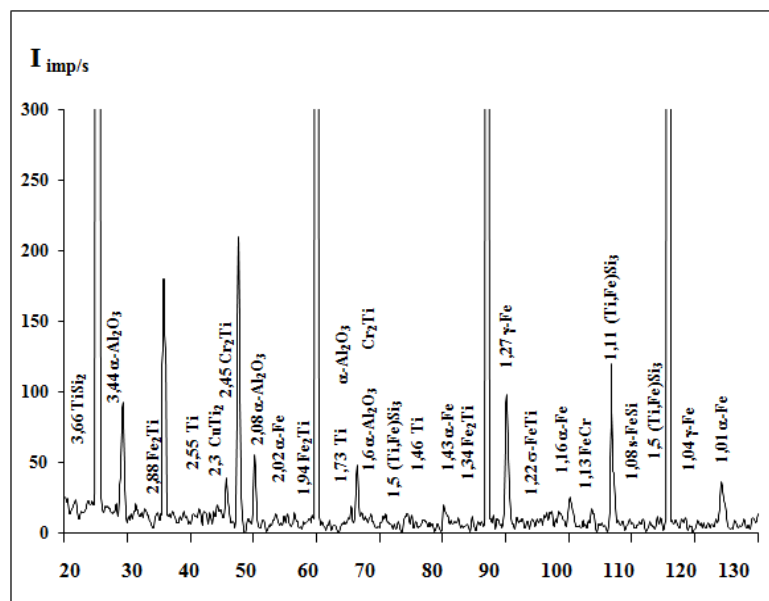


Fig. 3. X-ray diffraction pattern of CCCM sample with deposited protective layer

During the saturation of carbon materials with chromium, aluminum, and titanium, solid substitutional solutions are formed. The activation energy  $Q$  in  $\gamma$ -iron exceeds 250,8 kJ/mol, which, under non-stationary process conditions, enables not only the achievement of elevated temperatures but also a higher concentration of diffusing elements at the surface of the treated material.

Protective coatings synthesized under isothermal exposure conditions tend to exhibit elevated porosity levels and are commonly associated with the formation of the FeAl intermetallic phase. This phase, due to its structural characteristics, contributes to the penetration of oxygen into the substrate material, thereby reducing the protective efficacy of the coating. Conversely, coatings produced through the application of functionally active charges (FACs) display markedly improved thermal stability. This enhancement is primarily attributed to the increased incorporation of chromium, aluminum, silicon, and titanium within the coating matrix, which collectively facilitate the development of dense and adherent oxide films such as  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3$  — known for their thermodynamic stability at elevated temperatures.

A distinctive aspect of the high-temperature behavior of FAC-derived coatings lies in the sequential formation mechanism of protective sublayers. Initially, a chromium-aluminum diffusion coating is established, which subsequently undergoes further alloying with titanium, chromium, and silicon. This process leads to the emergence of thermally stable intermetallic compounds, including TiAl and  $\text{CrSi}_2$ , which significantly improve the coating's resistance to oxidative degradation under extreme thermal and environmental loads. The multistage architecture of such coatings contributes to their long-term integrity and functional performance in aggressive service conditions.

### Conclusions

X-ray diffraction and metallographic analyses of protective coatings applied to carbon-based substrates reveal that, across the entire range of temperature-time conditions investigated, these coatings consistently exhibit a dual-layered microstructure. The inner layer is predominantly composed of titanium carbide, while the outer layer's phase composition is governed by the alloying elements integrated into the functionally active charge. Specifically, when titanium is employed as an alloying constituent, the resultant coating comprises TiC alongside intermetallic phases such as  $\text{Al}_2\text{Cr}_3$ ,  $\text{CrAl}_2$ , and TiAl, imparting robust protective properties.

The factor experiment and regression analysis allowed us to build three-dimensional models of the dependence of heat resistance on the composition of the charge, confirming the optimality of the multistage mechanism of coating formation, where the primary CrAl layer is supplemented by high-temperature phases of TiAl and  $\text{CrSi}_2$ .



The incorporation of chromium, aluminum, and titanium facilitates the formation of stable substitutional solid solutions, with an activation energy in  $\gamma$ -iron exceeding 250.8 kJ/mol, which promotes efficient diffusion under non-isothermal conditions. In contrast, coatings synthesized via isothermal processing are characterized by elevated porosity and the presence of the FeAl phase, which compromises their integrity by permitting oxygen diffusion to the substrate surface.

A distinctive advantage of coatings produced using FAC lies in their staged formation mechanism. Initially, a CrAl-based protective layer is formed, followed by further alloying with titanium, and chromium, which leads to the development of high-temperature phases such as TiAl and CrSi<sub>2</sub>. This multistep process significantly enhances the thermal stability and environmental durability of the coatings under extreme service conditions.

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