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Investigation of the structural and phase state of detonation multilayer coatings based on NiCr/NiCr–Al_2O_3/Al_2O_3 during high-temperature oxidation

This paper presents a study of the structural and phase state of detonation multilayer coatings under high-temperature oxidation. The experiment aimed to find out the effect of high temperatures on the structural-phase state of coatings and their effectiveness in preventing oxidative processes. As a result of the research, it was found that after high-temperature tests, protective phases such as $NiCr_2O_4$, Al_2O_3, and Cr_2O_3 are formed in the structure of multilayer coatings. These phases were identified to play a key role in preventing intensive oxidation of the metal surface. It was determined that $NiCr_2O_4$ provides stability to the metal layer, and Al_2O_3 and Cr_2O_3 are effective barriers protecting the surface from aggressive environmental influences. It is important to note that no signs of fracture or detachment were found in the multilayer coatings during the entire experiment. After the first cycle, the uncoated sample experienced peeling of its oxide film, resulting in a significant loss of mass. It was determined that the uncoated steel experiences significantly higher mass loss, indicating a faster formation of oxides on its surface. The experimental data confirm the effectiveness of coatings in protecting against oxidative processes at high temperatures, thereby maintaining the stability and durability of the material under extreme thermal influences.

Keywords: detonation spraying, structure, phase composition, high-temperature oxidation, multilayer coatings.

Introduction

In modern industry, where components are exposed to harsh operating conditions, including high-temperature gas flows, aggressive chemical environments and abrasive particles, the issue of improving the wear resistance, heat resistance and corrosion resistance of material surfaces is becoming increasingly important [1-2]. To effectively solve this problem, the use of multilayer gradient coatings is recommended, which in recent decades have successfully found application in various industries such as power engineering and mechanical engineering [3-4].

Multilayer gradient coatings provide reliable protection of surfaces against wear, corrosion and oxidation, increasing the durability and performance of parts and reducing maintenance costs [5-6]. Their uniqueness lies in their ability to combine different material characteristics in a single coating due to smoothly varying composition along the thickness. Compositions consisting of metal-ceramic layers with a uniformly distributed oxide component in them are the most promising for harsh operating conditions. Recent research has led to multilayer gradient coatings combining nickel chromium (NiCr) and aluminum oxide (Al_2O_3), which showed outstanding properties and provided unique advantages over conventional homogeneous coatings [7–9]. Al_2O_3 stands out for its abrasion and corrosion resistance, excellent dielectric resistance and thermal shock resistance. These characteristics make Al_2O_3 coatings particularly attractive for surface protection of metal components that are exposed to harsh environments [9]. Scientific studies emphasize the importance of proper chemical composition selection in the interface between the substrate and the coating to ensure high corrosion resistance, indicating that the boundaries between layers can act as effective protective barriers in aggressive environments [10–14]. Studies [15] have confirmed that the use of nickel chromium as the bottom metal layer in multilayer coatings helps to improve the thermal resistance of oxide coatings. This is due to improved adhesion strength and equalization of the difference in thermal expansion coefficients between the metal sample and the coating. This combination minimizes the stresses generated during thermal cycling and therefore reduces the likelihood of coating failure.

Under conditions of cyclic oxidation and high temperatures, a special approach is required to protect surfaces from aggressive environments. There are many coating methods available, and the selection of an appropriate method plays a key role in ensuring the durability and effectiveness of the protection. From the analysis of literature studies, it is revealed that detonation sputtering is a promising technology for NiCr-Al_2O_3-based multilayer gradient coatings [16–19]. This method provides high sputtering speed and is capable of producing high density coatings, minimizing the formation of pores and defects in the coating structure. This approach can provide reduced porosity and increased corrosion resistance, which is critical in corrosive environments. Nevertheless, not all multilayer gradient coatings exhibit resistance to high temperatures under real operating conditions. In certain instances, structural degradation occurs due to exposure to high temperatures and cyclic thermal loads, potentially resulting in a loss of effectiveness of protective properties. Hence, to achieve maximum efficiency and durability of multilayer gradient coatings, it is crucial to conduct research focused on optimizing their chemical composition, structure, and application methods. This will enable us to enhance the technology for creating such coatings, rendering them more stable and effective in high temperatures and aggressive environments.

Hence, the purpose of our study is to investigate the structural and phase states of detonation multilayer coatings under conditions of high-temperature oxidation. To accomplish this objective, we intend to conduct a study on the structural and phase characteristics of multilayer coatings under high-temperature exposure.

Materials and methods of research.

NiCr, NiCr–Al_2O_3 and Al_2O_3 powders with an average size of 40–45 μm were used to apply multilayer coatings on stainless steel 12Kh18N10T samples. The sputtering was carried out using a CCDS2000 machine [13]. The use of detonation spraying technology provided an opportunity to create multilayer gradient coatings. This process allows to sequentially apply layers with changing composition, which leads to a coating with a gradual change in characteristics from layer to layer. Technological parameters of the process and the effect of detonation sputtering on the properties and phase-structural states of NiCr/NiCr–Al_2O_3/Al_2O_3-based coatings are described in detail in the scientific paper [17-18]. As a result of the positive conclusions obtained in previous studies [11, 17], further high-temperature tests were carried out exclusively on samples with five-layer coatings.

An X'Pert PRO X-ray diffractometer (using CuKα radiation) was used to study the structural-phase composition of the coatings. Imaging was carried out at the following parameters: tube voltage U = 40 kV; tube current I = 30 mA. The obtained diffractograms were decoded using the HighScore program. Investigations of surface microstructure and morphology of coating cross-section were carried out by scanning electron microscopy (SEM) on MIRA3 (Tescan, Czech Republic) with energy dispersive analysis attachment INCA ENERGY (“Oxford Instruments”, Great Britain) in the laboratory of Karaganda Buketov University.

Experiments on high-temperature oxidation were carried out at a temperature of 1100 °C using a muffle electric furnace model SNOL 3/1100. All tests were performed in accordance with GOST 6130-71 “Metals. Methods of determination of heat resistance”. This standard establishes a methodology for evaluating the resistance of metals to high temperatures. In this test, 50 cycles were performed, and each cycle included one hour of heating to 1100 °C, followed by a 20-minute cooling to room temperature. To assess the extent of corrosion, samples were periodically removed from the crucible and weighed on analytical scales to the nearest 0.1 mg to determine the change in mass. This allowed monitoring the effect of thermal cycling on the surface properties of the materials.

Results and discussion.

Two types of samples were tested to assess oxidation resistance: uncoated steel 12X18H10T and a multilayer coating based on NiCr/NiCr–Al_2O_3/Al_2O_3. The coatings were uniformly applied to all six sides of the samples to ensure uniformity and minimize measurement errors. Figure 1 shows the samples before and after the high-temperature oxidation tests.

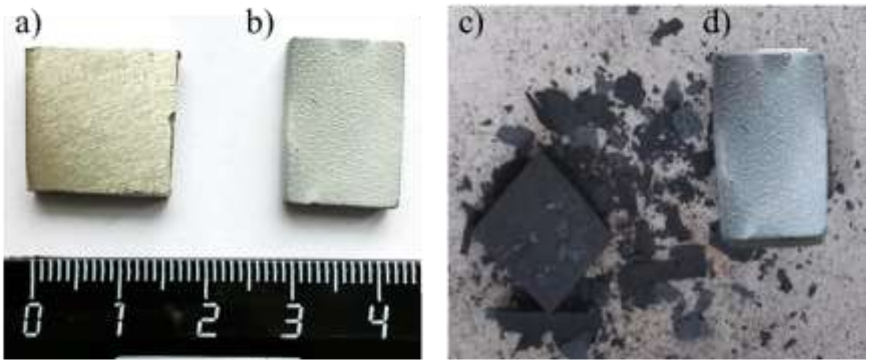


Figure 1. Samples before (a, b) and after (c, d) high-temperature oxidation tests

a-c) 12Kh18H10T steel; b-d) NiCr/NiCr–Al_2O_3/Al_2O_3 multilayer coating

Figure 2 shows the value of mass change of the samples after 50 cycles of high temperature oxidation test. The results indicated that the coated samples exhibited superior performance, whereas the uncoated samples yielded the poorest results. In the uncoated steel, intense delamination of the oxide film (scale) was already observed after the first cycle (Fig. 1c), and this process increased with time. The multilayer coatings showed no signs of failure or delamination throughout the experiment. It has been determined that the uncoated steel experiences a significantly higher mass loss, indicating a faster formation of oxides on its surface. Such a significant increase in the mass loss of the original sample may be due to the destruction of the sample due to cyclic exposure to high temperatures, which is not characteristic of coated samples.

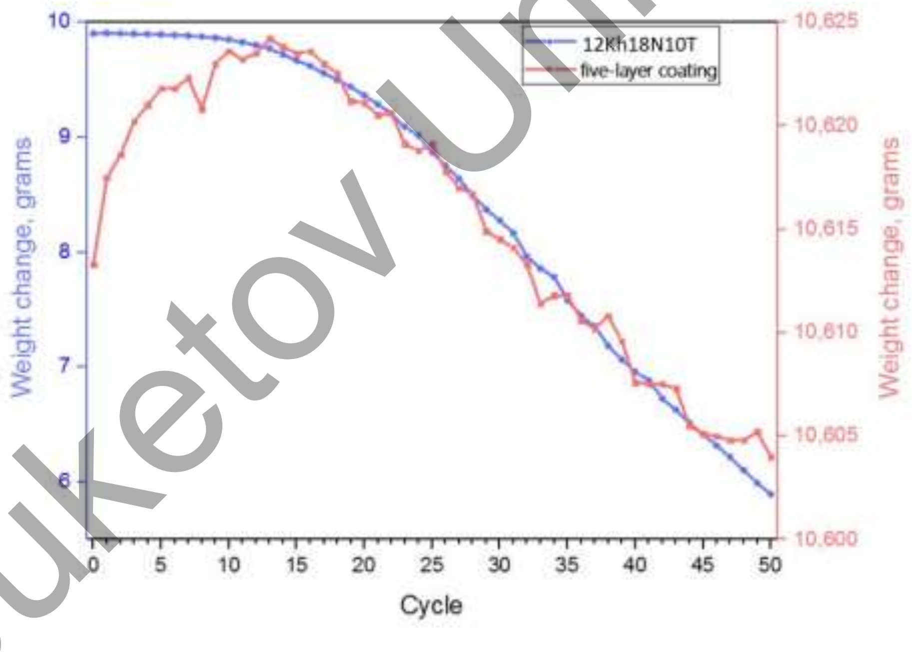


Figure 2. Weight change of uncoated steel and multilayer coating based on NiCr/NiCr–Al_2O_3/Al_2O_3 after high-temperature oxidation tests

Figure 3 shows the results of a scanning electron microscope of the cross-section of a multilayer coating NiCr/NiCr–Al_2O_3/Al_2O_3, after high-temperature tests for 50 hours at a temperature of 1100°C. After high-temperature oxidation tests, the multilayer coatings NiCr/NiCr–Al_2O_3/Al_2O_3 remain completely intact and dense. Cracks and peeling were not observed, which suggests that these multilayer coatings prevent direct contact of oxygen with the steel substrate and thus protect the steel from oxidation. The microstructure of the coating contains a significant amount of oxides and has low porosity.

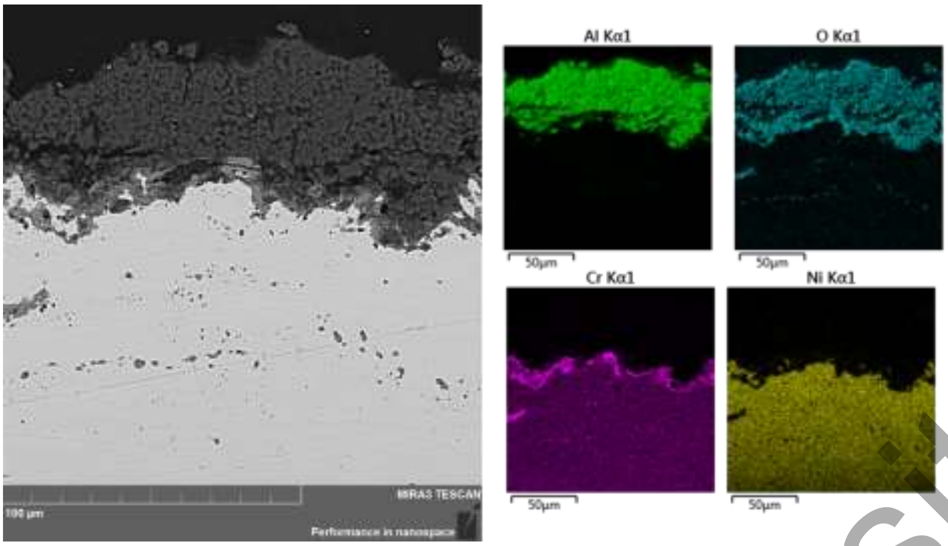


Figure 3. SEM images of cross-sectional morphology of multilayer coating after high-temperature oxidation

As a result of morphological studies of the cross-sectional structure of uncoated steel, it was found that heating to 1100 °C with prolonged holding time leads to the formation of a thin oxide layer (Fig. 4). This layer has a clear boundary with the metal, but its thickness is not uniform across the cross-section of the sample. The non-uniformity of the layer thickness across the cross-section of the sample may indicate complex oxidation processes and interaction with the environment in different parts of the material. Such morphological changes may affect the mechanical and chemical properties of the material.

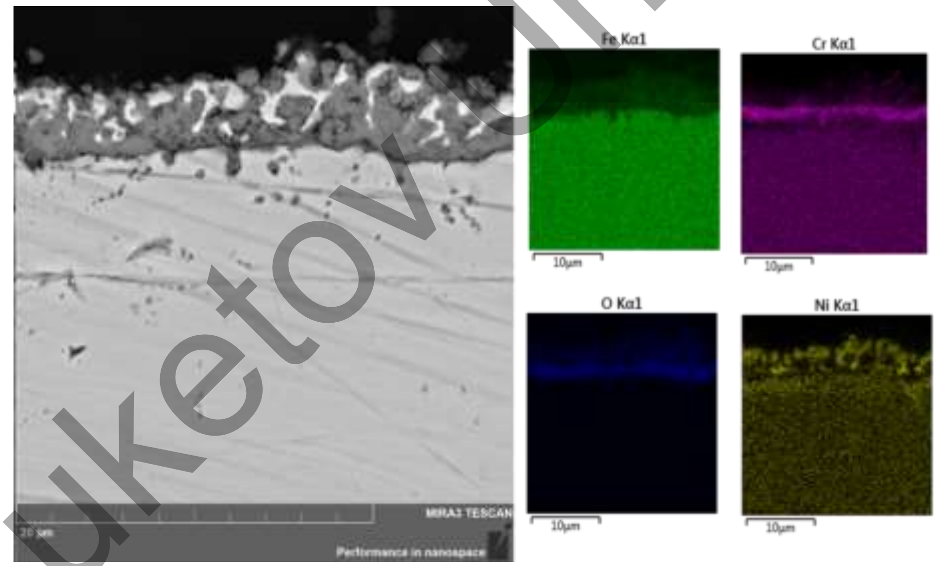
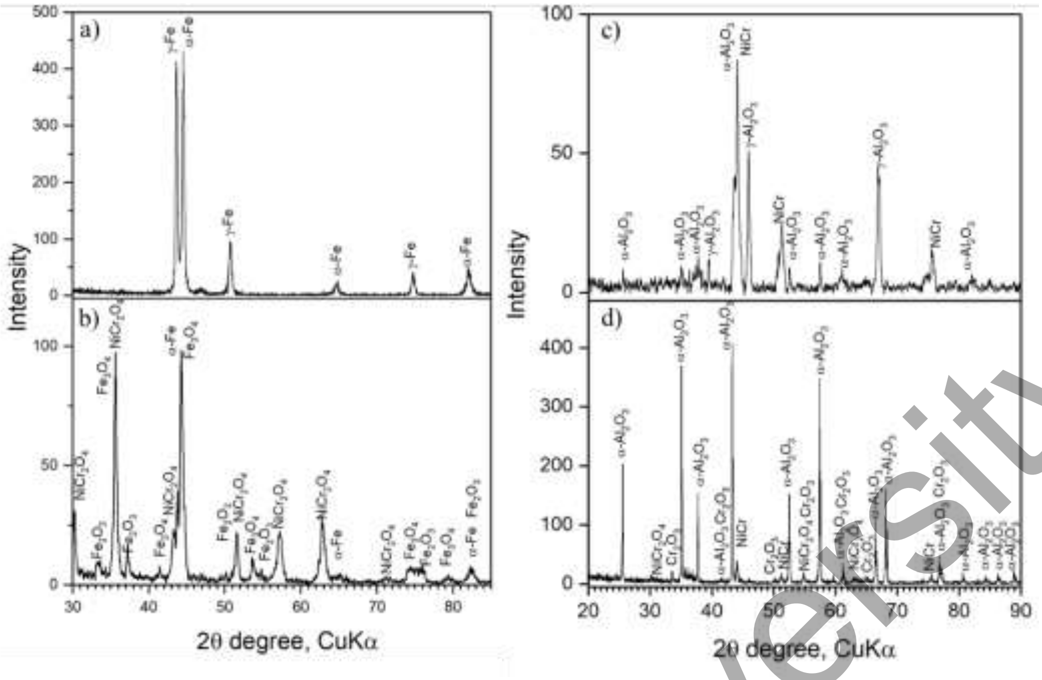


Figure 4. SEM images of cross-sectional morphology of uncoated steel after high-temperature oxidization

Figure 5 shows the X-ray images of the multilayer coatings before and after the high-temperature tests. It was found that the multilayer coatings consist of protective phases such as $NiCr_2O_4$, Al_2O_3 and Cr_2O_3 after high temperature testing. These phases play a key role in preventing intense oxidation at high temperatures. $NiCr_2O_4$ provides the stability of the metal layer, while Al_2O_3 and Cr_2O_3 are effective barriers protecting the surface from aggressive environmental influences. Prior to heat treatment, the graded five-layer coatings contain aluminum oxide phases including α- and γ-phases (Fig. 5c). Studies have shown that when heated to a temperature of 1100°C, γ-Al_2O_3 is converted to the more resistant α-Al_2O_3. This phase transition process from γ-phase to α-phase observed at high temperatures has also been reported in previous research studies [9, 20]. This phase transformation indicates the structural stability and adaptability of the coatings to changes in temperature conditions.



a-b) 12Kh18H10T steel; c-d) NiCr/NiCr–Al_2O_3/Al_2O_3 multilayer coating
Figure 5. Results of X-ray diffraction analysis before (a, c) and after (b, d) high temperature tests

After the experiment, uncoated steel shows the formation of different phases such as Fe_2O_3, Fe_3O_4 and $NiCr_2O_4$ (Fig. 5 b). This indicates an initiated oxidation process of the material under the influence of high temperatures. Especially interesting is the formation of $NiCr_2O_4$ spinel, which may be a consequence of high temperatures reaching 1100 °C. This process leads to the formation of a clear boundary between metal and oxide. The explanation of this effect is related to the partial dissolution of chromium carbides, which are oriented along the grain boundaries of the material.

Conclusion

Based on the conducted studies using five-layer coatings on 12Kh18N10T steel at high temperatures, the following conclusions can be made:

NiCr/NiCr–Al_2O_3/Al_2O_3 based multilayer coatings shown that the coated samples exhibit higher oxidation resistance characteristics compared to the uncoated sample. It is important to note that the multilayer coatings show no signs of failure or delamination throughout the experiment. In the uncoated sample, intense delamination of the oxide film was observed after the first cycle, resulting in significant mass loss. X-ray studies showed that the multilayer coatings contain protective phases such as $NiCr_2O_4$, Al_2O_3 and Cr_2O_3 after high temperature testing. It is found that these phases play a key role and indicates the effectiveness of these components in preventing the intense oxidation of the metal surface. It is determined that morphological changes such as non-uniformity of the oxide layer thickness can affect the mechanical properties of the material.

Thus, the experimental data confirm the effectiveness of coatings in protecting against oxidative processes at high temperatures and shows their importance in maintaining the stability and durability of the material under conditions of extreme thermal effects.

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Жоғары температуралы тотығу кезінде $NiCr/NiCr–Al_2O_3/Al_2O_3$ негізіндегі детонациялық көп қабатты жабындардың құрылымдық-фазалық күйін зерттеу

Жұмыста жоғары температуралы тотығу (оксидтеу) жағдайында детонациялық бүрку әдісі мен алынған көп қабатты жабындардың құрылымдық-фазалық күйінде болатын өзерістер бойынша зерттеу нәтижелері ұсынылған. Зерттеу жұмысының негізгі мақсаты детонациялық жабындардың құрылымдық-фазалық күйіне жоғары температураның әсері және олардың тотығу процестерінің алдын алудағы тиімділігін анықтау. Зерттеу жұмысының нәтижесінде жоғары температуралық сынақтардан кейін детонациялық көп қабатты жабындардың құрылымында $NiCr_2O_4$, Al_2O_3 және Cr_2O_3 сияқты қорғаныс фазаларының түзілетіні анықталды, сондай-ақ бұл фазалар металл бетінің қарқынды тотығуын болдырмауда шешуші рөл атқаратыны белгілі болды. $NiCr_2O_4$ фазасы металл қабатының тұрақтылығын қамтамасыз етуші екені айқындалды, ал Al_2O_3 және Cr_2O_3 бетті қоршаған ортаның агрессивті әсерінен қорғайтын тиімді кедергілер ретінде әрекет етеді. Көп қабатты жабындарда зерттеу жұмысының барысында бұзылу немесе қабыршақтану сияқты белгілердің байқалмағанын атап өту маңызды. Керісінше қапталмаған үлгі (12Х18Н10Т болаты) үшін бірінші циклден кейін оксидті пленканың бөлінуі байқалды, бұл массаның айтарлықтай жоғалуына (төмендеуіне) әкелді. Бастапқы болат массасының жоғалуы айтарлықтай жоғары екендігі анықталды, бұл оның бетінде оксидтердің тезірек түзілуін көрсетеді. Осылайша, зерттеу нәтижелері детонациялық көп қабатты жабындардың жоғары температура кезінде тотығу процестерінен қорғауда тиімді екенін растайды және де экстремалды жылу әсерлері жағдайында материалдың тұрақтылығы мен беріктігін сақтауда маңыздылыққа ие екенін көрсетеді.

Кілт сөздер: детонациялық бүрку, құрылым, фазалық құрамы, жоғары температуралы тотығу, көп қабатты жабындар.

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Исследование структурно-фазового состояния детонационных многослойных покрытий на основе $NiCr/NiCr–Al_2O_3/Al_2O_3$ при высокотемпературном окислении

В работе представлены результаты исследования структурно-фазового состояния детонационных многослойных покрытий в условиях высокотемпературного окисления. Основная цель эксперимента — выяснить, как воздействие высоких температур влияет на состояние этих покрытий и их способность предотвращать окислительные процессы. В результате исследования было обнаружено, что после высокотемпературных испытаний в структуре детонационных многослойных покрытий формируются защитные фазы, такие как $NiCr_2O_4$, Al_2O_3 и Cr_2O_3. Было установлено, что эти фазы играют ключевую роль в предотвращении интенсивного окисления металлической поверхности. Особенно выделяется роль $NiCr_2O_4$, обеспечивающего устойчивость металлического слоя, и Al_2O_3 с Cr_2O_3, которые действуют как эффективные барьеры, защищающие поверхность от воздействия окружающей среды. Важно отметить, что в многослойных покрытиях не обнаружено признаков разрушения или отслоения в течение всего эксперимента. В отличие от этого, в образце без покрытия (сталь 12Х18Н01Т) уже после первого цикла наблюдалось отслоение оксидной пленки, что существенно увеличило потерю массы. Определено, что потеря массы у стали без покрытия значительно выше, что указывает на более быстрое образование оксидов на её поверхности. Таким образом, результаты исследования подтверждают эффективность детонационных многослойных покрытий в защите от окислительных процессов при высоких температурах, а также указывают на то, что они играют важную роль в поддержании стабильности и прочности материала в условиях экстремальных тепловых эффектов.

Ключевые слова: детонационное напыление, структура, фазовый состав, высокотемпературное окисление, многослойные покрытия.

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