CHEMICAL TECHNOLOGIES, MATERIALS SCIENCES, METALLURGY
ХИМИЧЕСКИЕ ТЕХНОЛОГИИ, НАУКИ О МАТЕРИАЛАХ, МЕТАЛЛУРГИЯ

UDC 621.793.5
https://doi.org/10.23947/2541-9129-2025-9-2-170-178

Original Empirical Research

EDN: WDDMOV

Influence of Pre-Carburization on the Structure and Properties of Chromium Coatings on Steels Formed by Diffusion Alloying in Liquid Metal Media Solutions

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Abstract
Introduction. Diffusion alloying from the medium of low-melting liquid metal solutions (DALMMS) allows us to form coatings for metal products. At the same time, the carbon content in the alloy is reduced under the carbide coating layer, which negatively affects the operation of products under contact stresses. To neutralize decarburization, as well as to obtain deep hardened layers, a complex chemical-thermal treatment (CCTT) is proposed. It means pre-carburization and subsequent DALMMS with chromium. It is important to compare the characteristics of coatings on metal samples that have undergone and have not undergone carburization. The results of such studies have not been published before. The aim of the work is to analyze the effect of pre-carburization on chromium-based diffusion coatings and the structure of the coated sample.
Materials and Methods. The coatings were obtained by immersing St3 and 40X steel samples in a PbLi reaction transport medium with the addition of chromium. Some of the samples were previously subjected to vacuum carburization. The coating thickness and structure of the coated sample were determined using a universal microscope NU-2E (Carl Zeiss Jena). Electron microprobe analysis was performed on a Tescan Lyra 3 electron microscope with the Oxford Ultim MAX PCMA system. Microhardness was determined by the Dura Scan Falcon 500 microhardness tester. X-ray phase analysis (XPA) was performed on a Bruker D8 Advance Eco X-ray diffractometer.
Results. Without carburization, a coating with a thickness of 12 μm was formed on the St3 steel sample, while with carburization it was 22 μm. The difference was 1.83 times. The chromium diffusion depth in the sample without carburization was 18 μm. In the sample with pre-carburization it was 34 μm. Carburization provided a significant increase in the depth of the hardened layer. Without pre-treatment, the microhardness values of the coating were recorded after DALMMS: 1400 HV0.02 for Ct3 and 1650 HV0.02 for 40X. After CCTT: 1500 HV0.02 for Ct3 and 1800 HV0.0 for 40X. However, at a depth of 10 μm, the microhardness (160 HV0.02) was lower than that of the coated material for both samples. After CCTT, the areas with reduced microhardness disappeared, and the depth of the hardened layer was 1.5 mm for Ct3 and 2 mm for 40X.
Discussion and Conclusion. Pre-carburization helps to avoid the formation of a softened sublayer between the coating and the coated material, which is important for the performance of products under contact stresses. Consequently, chrome-coated structural steel parts can be used after carburization in conditions of abrasive corrosion and high mechanical loads. Examples of these applications include compressor equipment and oil and gas equipment.

Keywords: chemical-thermal strengthening methods, diffusion metallization, pre-carburization, diffusion alloying with chromium, decarburized ferrite sublayer

Acknowledgements. The authors would like to express their gratitude to the team of the Department of Materials Science and Composite Materials, Volgograd State Technical University, in particular to Bogdanov A.I., Cand.Sci. (Engineering), Associate Professor, for their assistance in obtaining the results.

For Citation. Svistun LI, Bobylyov EE, Nirov AD, Storozhenko ID, Popov RA. Influence of Pre-Carburization on the Structure and Properties of Chromium Coatings on Steels Formed by Diffusion Alloying in Liquid Metal Media Solutions. *Safety of Technogenic and Natural Systems.* 2025;9(2):170–178. https://doi.org/10.23947/2541-9129-2025-9-2-170-178

Оригинальное эмпирическое исследование

Влияние предварительной цементации на структуру и свойства диффузионных покрытий на основе хрома на сталях, полученных в среде легкоплавких жидкометаллических растворов

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Аннотация
Введение. Диффузионная металлизация из среды легкоплавких жидкометаллических растворов (ДМЛЖР) позволяет формировать покрытия для изделий из металла. При этом под слоем карбидного покрытия сокращается содержание углерода в сплаве, что негативно сказывается на работе изделий в условиях контактных напряжений. Для нейтрализации обезуглероживания, а также получения глубоких упрочненных слоев предложена комплексная химико-термическая обработка (КХТО) — это предварительная цементация и последующая ДМЛЖР хромом. Важно сопоставить особенности покрытий на металлических образцах, прошедших и не прошедших цементацию. Результаты таких исследований ранее не публиковались. Цель работы — анализ влияния предварительной цементации на хромовые диффузионные покрытия и структуру покрываемого образца.
Материалы и методы. Покрытия получены путем погружения образцов из сталей Ст3 и 40Х в реакционно-транспортную среду PbLi с добавлением хрома. Часть образцов предварительно прошла вакуумную цементацию. На универсальном микроскопе NU-2E Carl Zeiss Jena («Карл Цейс Джина» (англ.)) определялись толщина покрытия и структура образца. Микрорентгеноспектральный анализ (МРСА) проводился на электронном микроскопе Tescan Lyra 3 («Тискан Лира 3» (англ.)) с системой PCMA Oxford Ultim MAX («Пи-си-эм-эй Оксфорд Ультим МАКС» (англ.)). Микротвердость определял микротвердомер Dura Scan Falcon 500 («Дюра Скан Фалькон 500» (англ.)). Рентгенофазовый анализ (РФА) проводили на рентгеновском дифрактометре Bruker D8 Advance Eco («Брюкер Ди-8 Эдванс Эко» (англ.)).
Результаты исследования. Без цементации на образце из стали Ст3 сформировалось покрытие толщиной 12 мкм, с цементацией — 22 мкм. Разница — в 1,83 раза. Глубина диффузии хрома в образце без цементации составила 18 мкм, в образце с предварительной цементацией — 34 мкм. Цементация обеспечила значительное увеличение глубины упрочненного слоя. Без предварительной обработки после ДМЛЖР фиксировались показатели микротвердости покрытия: 1400 HV0,02 для Ст3 и 1650 HV0,02 для 40Х. После КХТО: 1500 HV0,02 для Ст3 и 1800 HV0,0 для 40Х. Однако на глубине 10 мкм микротвердость (160 HV0,02) оказалась ниже показателя покрываемого материала для обоих образцов. После КХТО исчезают зоны с пониженной микротвердостью, глубина упрочненного слоя — 1,5 мм для Ст3 и 2 мм для 40Х.
Обсуждение и заключение. Предварительная цементация позволяет избежать формирования разупрочненного подслоя между покрытием и покрываемым материалом, что важно для эксплуатации изделий при контактных напряжениях. Следовательно, детали из конструкционных сталей с хромовым покрытием после цементации можно использовать в условиях абразивно-коррозионного воздействия и высоких механических нагрузок. Примеры такой эксплуатации — компрессорная техника и нефтегазовое оборудование.

Ключевые слова: химико-термические методы упрочнения, диффузионная металлизация, предварительная цементация, диффузионное легирование хромом, обезуглероженный ферритный подслой

Благодарности. За помощь в получении результатов авторы благодарят сотрудников кафедры «Материаловедение и композиционные материалы» ФГБОУ ВО «Волгоградский государственный технический университет» и лично доцента, кандидата технических наук А.И. Богданова.

Для цитирования. Свистун Л.И., Бобылёв Э.Э., Ниров А.Д., Стороженко И.Д., Попов Р.А. Влияние предварительной цементации на структуру и свойства диффузионных покрытий на основе хрома на сталях, полученных в среде легкоплавких жидкометаллических растворов. *Безопасность техногенных и природных систем.* 2025;9(2):170–178. https://doi.org/10.23947/2541-9129-2025-9-2-170-178

Introduction. In modern mechanical engineering, surface hardening of parts is a widely used technique [1]. Known technologies make it possible to obtain products with different properties and structures [2] by changing the properties and structure of the surface layer [3]. Special attention should be paid to chemical and thermal hardening methods. They are characterized by simplicity [4], provide high-quality hardened surfaces [5], as well as a smooth change in structure and properties from coating to coated material [6]. The technology of diffusion alloying from the medium of low-melting liquid metal solutions (DALMMS) refers to the technologies of chemical and thermal treatment (CCTT) and is promising from the point of view of obtaining functional coatings [6]. This technology is used to form coatings based on Cr, Ti, W, Mo, Ni, Cu, etc. on the surface of products made of steels [6], hard alloys [7], and cast iron [8].

Chromium is one of the elements that is often used as a basis for functional coatings [9]. These coatings increase wear resistance of parts [10] and corrosion resistance in aggressive environments [11]. Chrome coatings can also improve resistance to simultaneous complex effects of mechanical and corrosive wear [12]. The high resistance to wear and corrosion is due to the fact that the coatings are based on carbides [13]. During the formation of these carbides, carbon diffuses from the coated material. As a result, the carbon content in the alloy under the coating decreases, leading to the formation of decarbonization zones. This causes the carbide layer to press onto the coated part during operation [14]. To counteract the effect of decarburization and further harden the coated material, complex chemical and thermal treatment (CCTT) can be employed. It means the pre-carburization and diffusion alloying of samples with chromium in the medium of low-melting liquid metal solutions [15].

The aim of this research was to analyze the effect of pre-carburization on the formation of chromium-based diffusion coatings and the structure of the coated sample.

Materials and Methods. CCTT and DALMMS were performed on cylindrical samples with a diameter of 20 mm and a length of 30 mm. The samples were made of St3 and 40X steel (Table 1).

Table 1

Chemical composition of the studied materials

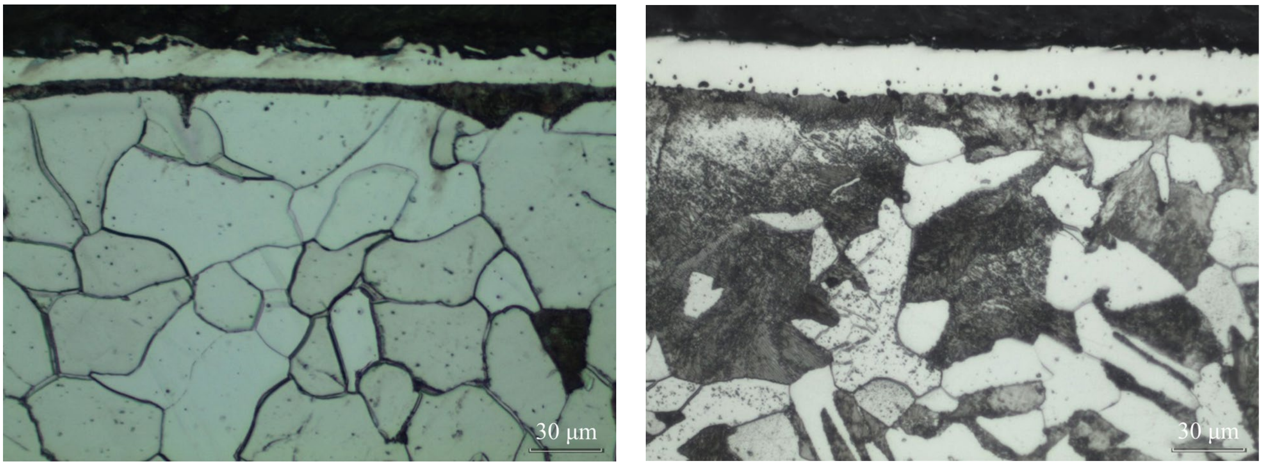
Steel grade	Element content, weight %							
	C	Si	Mn	Ni	S	P	Cr	Cu
St3	0.14–0.22	0.15–0.3	0.4–0.65	up to 0.3	up to 0.05	up to 0.04	up to 0.3	up to 0.3
40X	0.36–0.44	0.17–0.37	0.5–0.8	up to 0.3	up to 0.035	up to 0.035	0.8–1.1	up to 0.3

Chrome coatings were obtained through the process of diffusion alloying of samples in low-melting liquid metal solutions. To do this, we used the DALMMS setup [16]. The reaction transport medium was a PbLi eutectic melt, into which 10% chromium powder was added. The coating was applied by immersing and then isothermally exposing the samples in the melt at 1050°C for 8 hours, with argon filling the space above the melt. This process also involved solid-phase diffusion, leading to the formation of solid solutions and chemical compounds.

Prior to the start of DALMMS, in order to saturate the surface layers of steels with carbon, vacuum carburization was performed in a propane-butane mixture at a temperature of 950°C for 8 hours.

The structure of the coated material and the thickness of the coating were studied using a NU-2E universal optical microscope (Carl Zeiss Jena). The microhardness of the samples after DALMMS and CCTT was studied on a Dura Scan Falcon 500 microhardness tester. Electron microprobe analysis was performed on a Tescan Lyra 3 scanning electron microscope with the Oxford Ultim MAX PCMA system. An X-ray diffractometer Bruker D8 Advance Eco (Bruker AXS GmbH) with a θ-θ vertical goniometer was used for X-ray phase analysis (XPA). The samples were etched in a 4% alcohol solution of HNO_3.

Results. Figure 1 shows micrographs of St3 steel samples after DALMMS and CCTT.



a) *b)*

Fig. 1. Surface layers of St3 steel after 8-hour DALMMS at 1050°C:
a — without pre-carburization; *b* — with pre-carburization

Thus, pre-carburization affected both the steel and coating structure after DALMMS. Without pre-carburization, carbon diffused into the area under the coating, forming a pearlitic layer and a decarbonized ferritic layer in which there were no pearlitic grains. If pre-carburization was performed, the structure of the coated material changed. After CCTT, there was no decarbonized ferritic sublayer in it, even when low-carbon St3 steel was saturated. The structure of the material was perlite with ferrite inclusions (Fig. 1*b*).

Several zones could be distinguished that formed the surface layers of the material after CCTT:

– coating,

– transition zone between the coating and the material to be coated,

– carburization zone,

– transition carburization zone — base.

Figure 2 shows the structure of a 40X steel sample after CCTT.

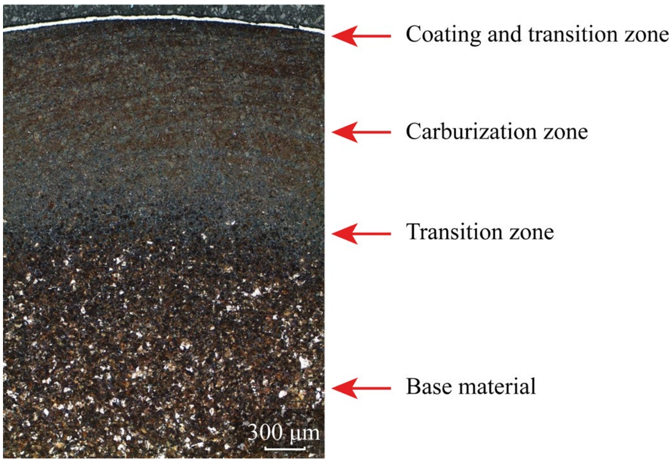
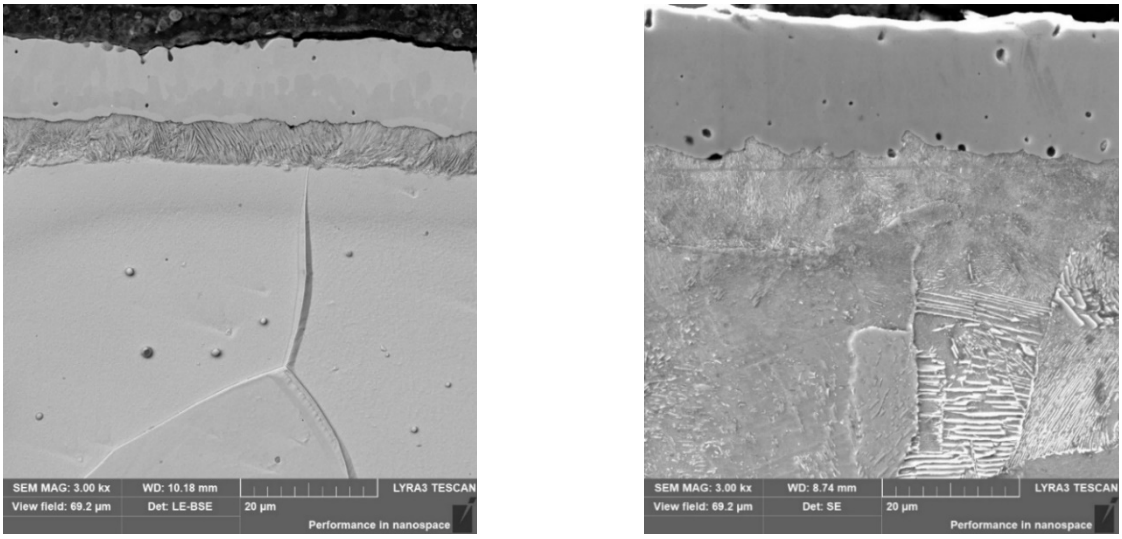


Fig. 2. Sample structure after CCTT

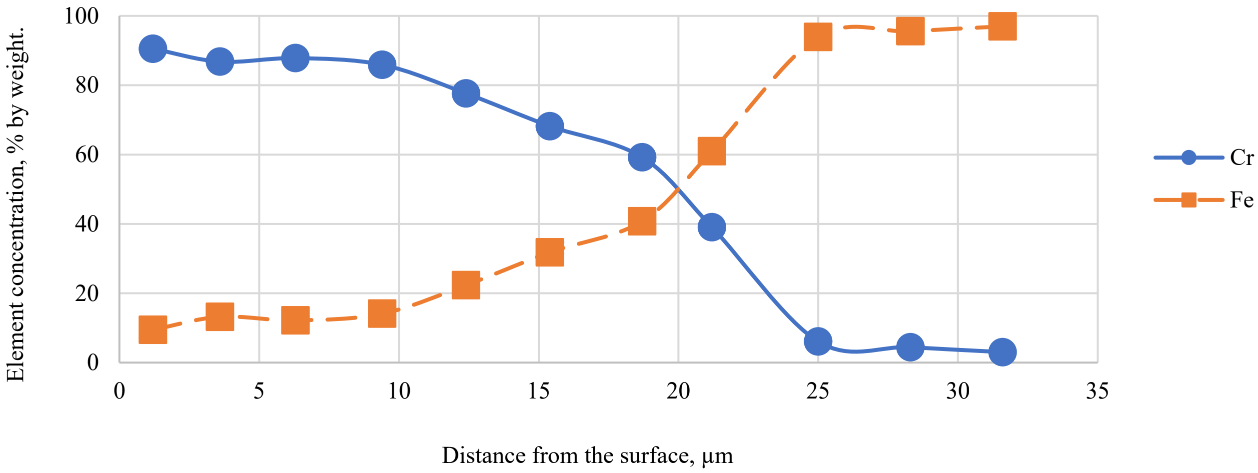
In case of DALMMS without pre-carburization, a zone of lamellar perlite alloyed with chromium was formed under the coating, followed by a soft ferritic layer. There was no decarbonized layer in the pretreated samples. Without carburization, a coating with a thickness of 12 μm was formed on the St3 steel sample; with carburization it was 22 μm. Thus, pre-carburization helped to increase the coating thickness by 1.83 times. There was a physical explanation for this result. Pre-carburization increased the intensity of carbon diffusion from the sample to the adsorbed chromium. Carbon heterodiffusion was recorded under the influence of high temperatures characteristic of DALMMS. This helped to equalize the carbon content and eliminate decarbonized zones that could occur as a result of the formation of carbides. Figure 3 shows micrographs of 40X steel after DALMMS and CCTT. On the sample after CCTT, the perlite structure was thinner, and there was no decarbonized zone.



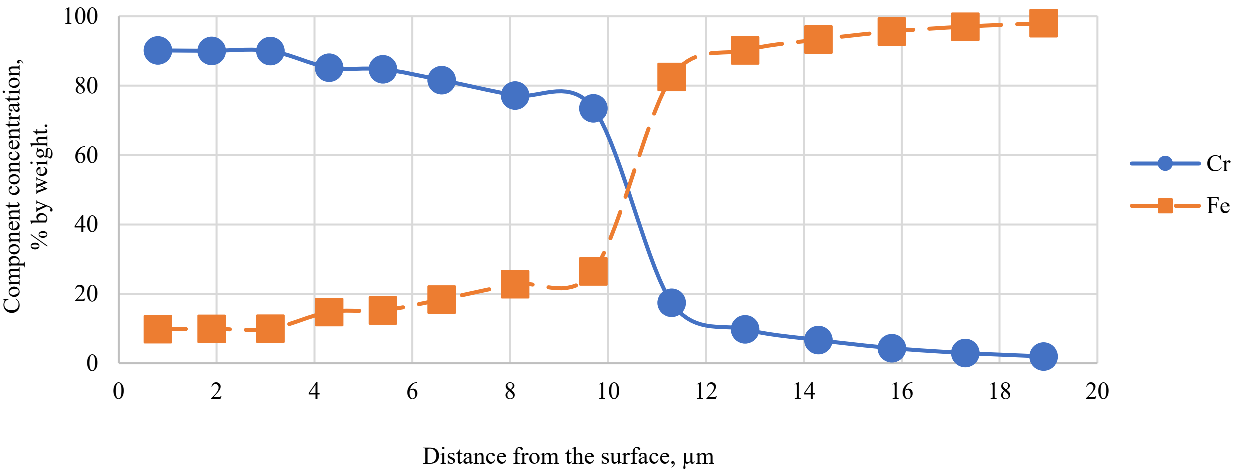
a) *b)*

Fig. 3. The structure of the 40X steel surface layer after an 8-hour DALMMS at 1050°C:
a — DALMMS; *b* — CCTT

Carburization did not significantly affect the chemical and phase composition. In both cases, the chromium content on the surface was 90% (wt.). However, the depth of chromium diffusion in the sample without carburization was 18 µm. In the sample with pre-carburization it was 34 µm, that is, twice as much. The results of electron microprobe analysis of the samples are shown in Figure 4.



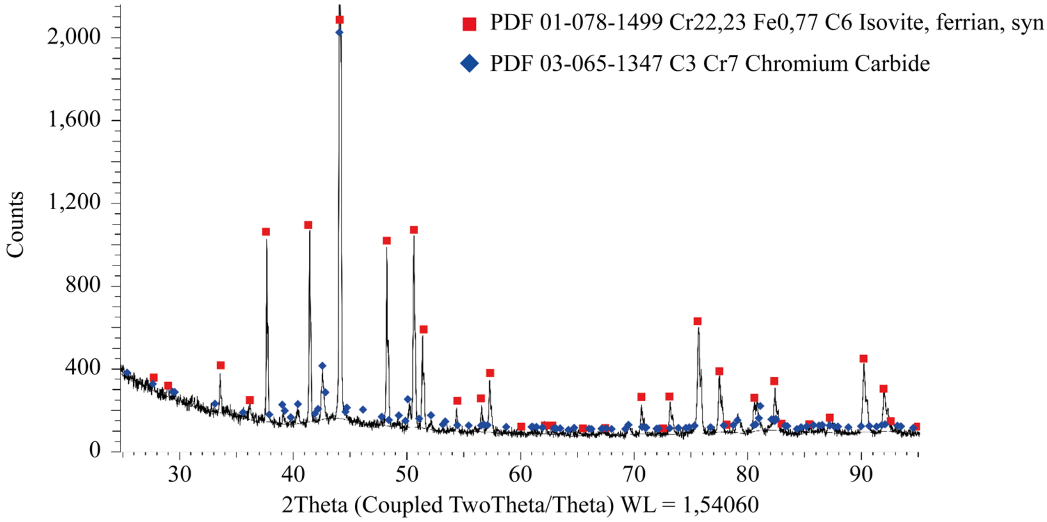
a)



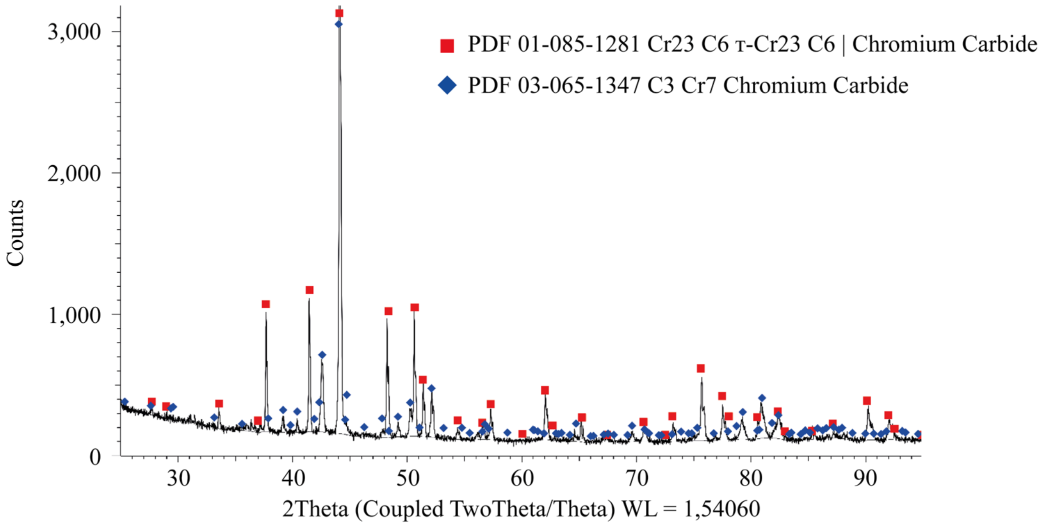
b)

Fig. 4. The results of electron microprobe analysis of coatings after an 8-hour DALMMS at 1050°C:
a — without pre-carburization; *b* — with pre-carburization

Phase composition of the coatings was represented by chromium carbides $M_{23}C_6$ and M_7C_3. In the sample without pre-carburization, there was a slight iron content in chromium carbide $M_{23}C_6$ (Fig. 5).



a)



b)

Fig. 5. The results of X-ray phase analysis of the coatings after 8-hour DALMMS at 1,050°C:
a — without pre-carburization; *b* — with pre-carburization

Pre-carburization provided a significant increase in the depth of the hardened layer (Fig. 6).

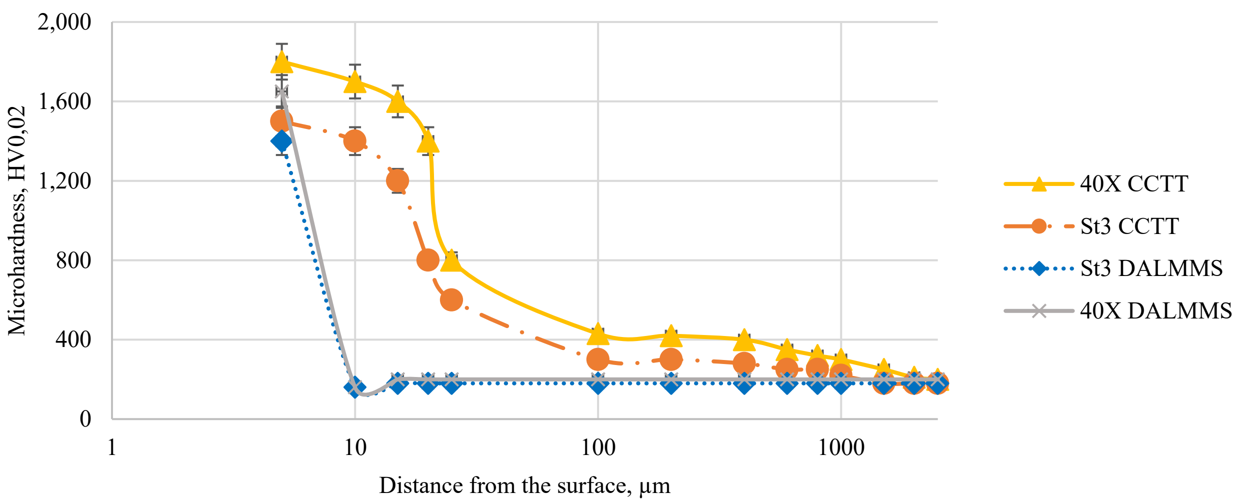


Fig. 6. Distribution of microhardness in samples after 8-hour DALMMS at 1050°C

According to Figure 6, on samples without pre-carburization, the microhardness index of the coating after DALMMS for St3 steel was 1400 $HV_{0,02}$, 40X — 1650 $HV_{0,02}$. After CCTT, the microhardness of the St3 steel coating was 1500 $HV_{0,02}$, 40X — 1800 $HV_{0,0}$. However, at a depth of 10 µm, the microhardness was 160 $HV_{0,02}$. This was lower than the microhardness of the coated material in both cases (i.e. for St3 and 40X samples). After CCTT, the microhardness distribution had a different character:

– there were no zones with reduced microhardness,

– the depth of the hardened layer was 1.5 mm for St3 steel and 2 mm for 40X steel.

Discussion and Conclusion. Thus, under the conditions considered, the main effect of carburization was manifested in the intensification of coating growth and the exclusion of the formation of a soft ferritic layer between the coating and the coated material. The composition of the coating and the chromium content did not depend on carburization. In any case, the coatings consisted of chromium carbides such as $M_{23}C_6$ and M_7C_3, the chromium content on the surface reached 90%.

CCTT affected the structure of the layer between the coating and the base material. In case of DALMMS, the layer under the coating had a pearlitic structure, turning into a ferritic one and further into the structure of the coated material. Pre-carburization made the transition zone more uniform. Here, a pearlitic structure was formed with a gradual change in chromium concentration — from 10% at the boundary of the coating and the pearlitic zone to 0.3% at a depth of 35 µm.

Thus, the positive results of pre-carburization have been proven. Firstly, the carbon content in the surface layers of the product increased, and this made it possible to intensify carburization.

Secondly, it became possible to obtain a carbon-saturated layer between the coating and the base material, which also strengthened the structure of the coated material. Therefore, it can be argued that pre-carburization is promising in terms of expanding the scope of application of parts with diffusion coatings based on chromium carbide. It eliminates the penetration of the layer under the coating and its subsequent destruction. The parts after CCTT are capable of operating at high contact stresses without chipping the functional layer.

Thirdly, the formation of a diffusion coating is accelerated. In addition, it will be thicker. As part of this study, the indicator was 1.83. The coating obtained after carburization was that much thicker (compared with the technology without pre- carburization). This indicates the significant role of carbon in the mechanism of coating creation.

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Claimed Contributorship:

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Conflict of Interest Statement: **the authors declare no conflict of interest.**

All authors have read and approved the final manuscript.

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Заявленный вклад авторов:
Л.И. Свистун: разработка концепции.
Э.Э. Бобылёв: рецензирование и редактирование рукописи.
А.Д. Ниров: написание черновика рукописи.
И.Д. Стороженко: проведение исследования.
Р.А. Попов: проведение исследования.

Конфликт интересов: **авторы заявляют об отсутствии конфликта интересов.**

Все авторы прочли и одобрили окончательный вариант рукописи.

Received / Поступила в редакцию 10.02.2025
Reviewed / Поступила после рецензирования 05.03.2025
Accepted / Принята к публикации 17.03.2025