ISSN 2075-8545 (online)

Nanotechnologies in construction
Нанотехнологии в строительстве

2026; 18 (5):
621–626

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Original article
https://doi.org/10.15828/2075-8545-2026-18-5-621-626

Creating a technology to coat the insulating material on the "cold" section of a cartridge-style electric heater for operation in aggressive environments

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ABSTRACT

Introduction. Currently, fluorine polymers have become quite widespread in various industries. One of the fields of application of fluoroplastic coatings in the oil industry is their use as protective coatings to prevent the deposition of paraffin plugs on the walls of tubing and field pipelines. Fluoroplastic parts find promising application in machines and units of oil refining and petrochemical industries, where high corrosion resistance, sufficient strength and high tribotechnical properties are required. The complexity of the ubiquity of fluoroplastic coatings is its low adhesion to the metal surface. In this regard, fluoroplastic coatings are typically complex systems comprising directly fluoroplastic polymer, binders and functional fillers. **Materials and methods.** The study used metal plates made of titanium alloy VT6 and carbon steel St3 coated with an aqueous fluoroplastic suspension. Fluoroplastic polymer coatings have poor adhesive strength to the metal surface on which they are applied. Therefore, in order to ensure a high level of adhesion of the coating to the metal substrate, it is necessary to use complex methods of preliminary surface preparation. The coating was applied to the pre-prepared surface of the metal plates by air spraying using a spray gun and compressed air. The key parameters for the formation of the fluoroplastic film were drying and sintering modes. Adhesion was evaluated by the methods of "X-shaped" notch (State Standard 32702.2-2014) and tearing in a tensile machine on cylinders (State Standard 32299-2025). **Results.** Proposed method comprises application of intermediate primer layer from suspension containing chromium (III) oxide, phosphoric acid, water and fluoroplastic. The intermediate adhesive primer layer was applied by air spraying under the same conditions as the base fluoroplastic coating. This ensured that the proposed drying and sintering conditions met the base layer. The use of a primer made it possible to realize a gradient transition from the metal substrate to the fluoroplastic layer and significantly increase the adhesion properties. **Conclusion.** The results show a significant improvement in adhesion compared to the control samples without primer: the tensile strength value by the tear method is 2.56 N/mm^2, and by the X-notch method - from 5 points to 1 point (traces of peeling or removal of the coating along the incisions). The method is easy to implement and promising for use in protective coatings.

KEYWORDS: fluoroplastic, adhesion, primer, chromium (III) oxide, phosphoric acid, "X-notching" method, tearing method

SOURCES OF FUNDING FOR THE SCIENTIFIC WORK THAT RESULTED IN THE PUBLICATION: This work was conducted as part of the Strategic Project "New technologies, technical, and intelligent systems in mechanical engineering" of the Ufa State Petroleum Technological University Development Program for 2021–2030 in accordance with the Priority 2030 Strategic Academic Leadership Program.

ACKNOWLEDGEMENTS: The authors are grateful for the financial support of this project.

FOR CITATION:
Latypov O.R., Kim K.V., Lukyanov V.V., Latypova D.R., Daminev R.R., Ahmadullina A.F. Creating a technology to coat the insulating material on the "cold" section of a cartridge-style electric heater for operation in aggressive environments. *Nanotechnologies in construction.* 2026;18(5):621–626. https://doi.org/10.15828/2075-8545-2026-18-5-621-626. – EDN: MYGYGS.

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ISSN 2075-8545 (online)

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Разработка технологии нанесения изолирующего материала на «холодную» часть патронного электрического нагревателя для эксплуатации в агрессивных средах

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АННОТАЦИЯ

Введение. В настоящий момент фторполимеры получили достаточно широкое распространение в различных отраслях промышленности. Одной из областей применения фторопластовых покрытий в нефтяной промышленности является использование их в качестве защитных покрытий для предотвращения отложения парафиновых пробок на стенках насосно-компрессорных труб и промысловых трубопроводов. Детали из фторопластов находят перспективное применение в машинах и агрегатах нефтеперерабатывающих и нефтехимических производств, где необходимы высокая коррозионная стойкость, достаточная прочность и высокие триботехнические свойства. Сложностью повсеместного распространения покрытий на основе фторопласта является его низкая адгезия к металлической поверхности. В связи с этим покрытия на основе фторопластов обычно представляют сложные системы, включающие непосредственно фторопластовый полимер, связующие добавки и функциональные наполнители. **Материалы и методы.** В исследовании использовались металлические пластины из титанового сплава ВТ6 и углеродистой стали Ст3 с нанесенным покрытием из водной фторопластовой суспензии с наноразмерными частицами. Фторопластовые полимерные покрытия имеют слабую адгезионную прочность к металлической поверхности, на которую они наносятся. Поэтому для обеспечения высокого уровня адгезии покрытия к металлической подложке необходимо применение комплексных методов предварительной подготовки поверхности. Покрытие наносилось на предварительно подготовленную поверхность металлических пластин методом пневматического распыления с использованием краскопульта и сжатого воздуха. Ключевыми параметрами формирования фторопластовой пленки являлись режимы сушки и спекания. Адгезия оценивалась по методам «Х-образного» надреза (ГОСТ 32702.2-2014) и отрыва в разрывной машине на цилиндрах (ГОСТ 32299-2025). **Результаты.** В работе предложен метод повышения адгезии фторопластовой пленки толщиной 500 мкм к металлической пластине путем нанесения промежуточного грунтовочного слоя из суспензии, содержащей оксид хрома (III), фосфорную кислоту, воду и фторопласт. Промежуточный адгезионный грунтовочный слой наносили пневмораспылением с теми же режимами, что и основное фторопластовое покрытие. За счет этого обеспечивалось соответствие предложенных условий сушки и спекания к основному слою. Применение грунтовки позволяло реализовать градиентный переход от металлической подложки к фторопластовому слою и значительно повысить адгезионные свойства. **Заключение.** Полученные результаты демонстрируют значительное улучшение адгезии по сравнению с контрольными образцами без грунтовки: значение предела прочности по методу отрыва равно 2,56 Н/мм^2, а по методу Х-надреза – с 5 баллов до 1 балла (следы отслаивания или удаления покрытия вдоль надрезов). Метод прост в реализации и перспективен для применения в защитных покрытиях.

КЛЮЧЕВЫЕ СЛОВА: фторопласт, адгезия, грунтовка, оксид хрома (III), фосфорная кислота, метод «Х- образного» надреза, метод отрыва

ИСТОЧНИКИ ФИНАНСИРОВАНИЯ НАУЧНОЙ РАБОТЫ, РЕЗУЛЬТАТОМ КОТОРОЙ СТАЛА ПУБЛИКАЦИЯ: Работа проводилась в рамках реализации Стратегического проекта «Новые технологии, технические и интеллектуальные системы в машиностроении» программы развития ФГБОУ ВО «Уфимский государственный нефтяной технический университет» на 2021–2030 годы в соответствии с программой стратегического академического лидерства «Приоритет 2030».

БЛАГОДАРНОСТИ: Авторы выражают благодарность за финансовую поддержку проекта.

ДЛЯ ЦИТИРОВАНИЯ:

Латыпов О.Р., Ким К.В., Лукьянов В.В., Латыпова Д.Р., Даминев Р.Р., Ахмадуллина А.Ф. Разработка технологии нанесения изолирующего материала на «холодную» часть патронного электрического нагревателя для эксплуатации в агрессивных средах. *Нанотехнологии в строительстве.* 2026;18(5):621–626. https://doi.org/10.15828/2075-8545-2026-18-5-621-626. – EDN: MYGYGS.

ISSN 2075-8545 (online)

INTRODUCTION

Polytetrafluoroethylene (fluoroplastic) is one of the most promising fluoropolymers used as protective coatings for metal structures. Due to its high chemical resistance, low coefficient of friction, wide temperature range of operation and dielectric properties, fluoroplastic films are used in petrochemical equipment, aircraft engineering, mechanical engineering and a number of other industries. At the same time, low surface energy and high crystallinity of polytetrafluoroethylene lead to its low adhesion to most materials, which requires special surface preparation and the use of appropriate primers or copolymer bonds [1]. In particular, the literature describes in detail the properties and features of the use of an intermediate layer based on gossypol resin [2], which demonstrates high compatibility with various paint materials. Most studies are devoted to modified epoxy compositions [3], which, due to their high adhesion, often serve as the basis for primers. In addition, studies are being conducted on polyaniline primer as a functional component of anti-corrosion coatings that can affect electrochemical processes at the phase interface [4]. In general, the use of primers and adhesive layers helps to significantly increase the service life of the coating, including in a corrosive environment, where this sublayer acts as additional protection against corrosion. In some cases, a system of a primer and an additional intermediate coating between the soil and the finish coating is used to protect the metal from chemically aggressive media.

A number of studies have shown that modification of the surface of fluoroplastic using plasma treatment, in particular atmospheric nitrogen plasma, leads to an increase in wettability, partial surface defluorination and the introduction of oxygen and nitrogen-containing functional groups, which improves adhesion characteristics compared to non-modified polymer [5, 6]. Similar effects are observed during ion-beam treatment of fluoroplastic films, when ion irradiation causes a change in the chemical composition of the upper layers, an improvement in metal adhesion to the modified surface of the polymer [7]. However, such methods require vacuum or plasma equipment and often focus on thin films and specific material pairs, which limits their use in mass processes. Along with the modification of the polymer itself, the preparation of the metal substrate plays an important role. Phosphating treatments are traditionally used to increase the adhesion of paint and polymer coatings to steels and galvanized steel due to the formation of a thin layer of metal phosphates on the surface [8, 9]. Reviews of conversion coatings emphasize that phosphate and chromate films act as an intermediate layer that increases the adhesion of subsequent coatings [10].

Work on phosphating and the use of phosphate primers shows that such conversion layers can significantly increase the adhesion of organic and polymer coatings to metal substrates. A number of studies have found that during preliminary phosphating of steel and galvanized surfaces, subsequent paint and polymer systems form a stronger and more stable connection with the substrate compared to unmodified surfaces, while the most pronounced effect is achieved in systems with epoxy, polyester binders and polymer films applied to the phosphated layer [11, 12].

The purpose of this work is to experimentally evaluate the adhesion of a 500 μm thick fluoroplastic film to a metal substrate using a primer containing fluoroplastic, chromium oxide (III) (Cr_2O_3) in an aqueous medium and phosphoric acid (H_3PO_4). This primer is aimed at the "metal-primer-fluoroplastic (film)" system and uses fluoroplastic directly in the primer layer, forming a gradient transition to the main coating.

MATERIALS AND METHODS

The study used metal plates made of titanium alloy VT6 (analogous to Ti–6Al–4V) and carbon steel St3, which had dimensions of 120×60×1 mm. Before coating, the plates were cleaned of contaminants and degreased with acetone xh. in an ultrasonic bath "ODA-MH280" (China) with a power of 1100 W at a frequency of 40 kHz for 10 minutes at a temperature of 20–22 °C. This operation removed surface organic contaminants, which is an important condition for forming a uniform and stable adhesive bond.

An aqueous fluoroplastic suspension manufactured by HaloPolymer Perm JSC was used as the main coating material. The coating was applied to pre-prepared plates by a spray method using a spray gun and compressed air. The working pressure of compressed air was 0.2–0.3 MPa, the distance from the nozzle to the treated surface was in the range of 300-400 mm, which ensured uniform formation of a monolayer of suspension without splashing and draining.

The key parameters for the formation of the fluoroplastic film were drying and sintering modes. Drying was carried out at a temperature not exceeding 90 °C for 45 minutes to complete the removal of free and bound moisture from the aqueous suspension, while the absence of cracks and bubbles in the dried layer was monitored. Sintering was carried out at a temperature of 370 °C for 8–10 minutes, followed by cooling in an oven to room temperature. To achieve a given coating thickness (500 μm), the process was repeated repeatedly in sequence, ensuring the accumulation of layers with control of their thickness and continuity. All drying and sintering operations were carried out in the laboratory electric furnace "KS-18" (Russia), equipped with thermal control and uniform temperature distribution over the working volume.

The adhesion strength of the formed fluoroplastic films was evaluated using two standard methods: the

ISSN 2075-8545 (online)

X-cut method according to State Standard 32702.2-2014 and the normal tear method according to State Standard 32299-2025 on samples with dimensions of 50×50×1 mm. Due to the pronounced release properties and the difficulty of gluing the bursting cylinders to the surface of the fluoroplastic film, the use of classical methods for assessing tear strength has proven difficult. In this regard, a modified technique has been developed that allows measurements to be carried out without compromising the integrity of the coating. Films were formed on the end faces of the pair of plates, contacted and thermoplastic bonded at 330 °C (near the melting point of the fluoroplastic) for 5–7 minutes. As a result, a continuous layer of fluoroplastic serving as an adhesive seam was formed between the ends. Metal bursting cylinders fixed with epoxy rubber adhesive KDS-19 were mounted on the thus prepared glued plates. Adhesion tests by the tearing method were carried out on the universal test machine "REM-U-A" (Russia) at a fixed loading speed. The scheme of the proposed procedure is shown in Fig. 1.

A number of studies have shown that in order to improve the adhesive strength of fluoroplastic films, it is advisable for kmetallic substrates to pre-apply an intermediate adhesive layer. In this work, as such an intermediate layer, a suspension containing fluoroplast, chromium (III) oxide (Cr_2O_3) in an aqueous medium and phosphoric acid (H_3PO_4) was used. This intermediate coating formed a thin primer layer applied by air spraying under the same conditions as the base fluoroplastic coating, whereby the drying and sintering conditions of the base layer were satisfied. The use of a primer made it possible to realize a gradient transition from a metal substrate to a fluoroplastic layer and improve adhesion properties.

RESULTS AND DISCUSSION

When evaluating the adhesion of fluoroplastic films with a thickness of 500 μm using the "X-shaped" notch method [13], a significant difference was found in the behavior of coatings on samples with and without primer. For films formed without a preliminary primer layer, intense and continuous peeling of the coating around the perimeter of the notch was observed. In the visual assessment on a five-point scale, this corresponded to 5 points. Which indicates the complete absence of adhesion of fluoroplastic films to the metal substrate under direct contact conditions. For films applied with a primer layer, delamination along the perimeter of the notch was expressed to a minimal extent. In most cases, only local, fragmentary flaking was observed, with a visual score corresponding to 1 point. This indicates a significant improvement in adhesion strength and the formation of a sufficiently stable joint capable of withstanding the mechanical effects applied by the X-notch method.

When evaluating adhesion by the normal tearing method using the developed method, it was found that the absence of a primer layer leads to systematic defects in the formation of an adhesive seam. For films formed from a fluoroplastic slurry without primer and subsequent thermoplastic bonding of the plate end faces, spontaneous peeling of the coating from both ends was observed. The defect manifested itself in the form of separation of the fluoroplastic film at the border of contact with the ends of the metal plates, which led to the destruction of the integrity of the "metal-fluoroplastic (film)" system. As a result, such samples could not be used to quantify the peel adhesion strength because the

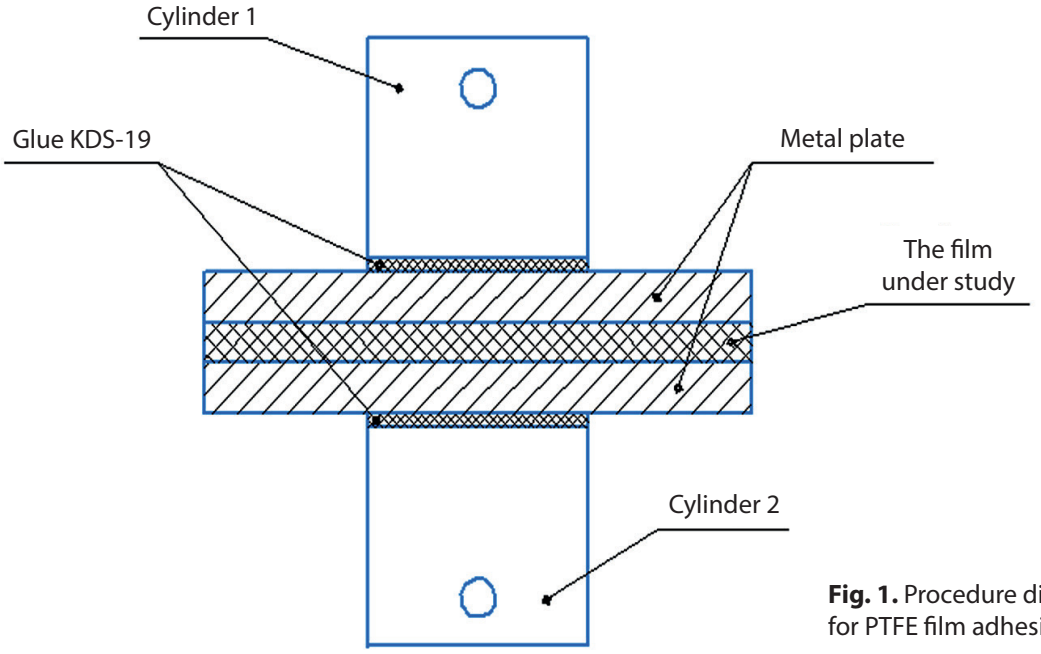

Fig. 1. Procedure diagram for PTFE film adhesion test

ISSN 2075-8545 (online)

Fig. 2. Appearance of coating with primer layer after normal tearing test (modified method)

failure occurred during the preparation stage and was not due to external loading.

For films formed using a primer layer, the adhesive seam at the ends of the plates retained integrity, and the destruction occurred under the influence of an external load on the test machine (Fig. 2).

A series of tests on the metal-soil-fluoroplastic (film) system resulted in the average values presented in Table 1.

These values indicate the formation of a physically correct and stable adhesive joint, in which the fracture occurs cohesively within the fluoroplastic matrix or adjacent zone, and not in the form of spontaneous detachment of the coating from the substrate.

Table 1. Peel adhesion test results

Maximum breaking load, N	Maximum deformation, mm	Tensile strength, N/mm²
1807.35	4.39	2.56

CONCLUSION

1. Application of a primer layer from a suspension containing fluoroplastic, chromium oxide (III) (Cr_2O_3) in aqueous medium and phosphoric acid (H_3PO_4), increases the adhesion strength of a fluoroplastic film with a thickness of 500 microns to metal substrates, which is confirmed by both visual assessment by the method of "X-shaped" notch and quantitative data by the method of normal separation. In a system with a primer layer, the degree of delamination is reduced to 1 point according to State Standard 32702.2-2014, and stable values are recorded during the test for the separation of registration under load: the average maximum load is 1807.35 N. The maximum deformation is 4.39 mm, the ultimate strength is 2.56 N/mm², which indicates the formation of a physically correct adhesive joint.

2. The mechanism of increasing adhesion is associated with the complex effect of the phosphate oxide layer and the gradient transition. Phosphoric acid, together with chromium oxide, forms a porous structure on the metal surface with a developed actual contact area, improving mechanical engagement with subsequent polymer and fluoroplastic coatings. The presence of fluoroplastic in the primer provides a gradient transition from the metal substrate through the intermediate layer to the main fluoroplastic film, which reduces the jump in physical and mechanical properties and reduces the concentration of stresses in the contact zone.

3. The results obtained demonstrate that the developed primer suspension can be recommended for use in technologies for applying fluoroplastic coatings to metal structures, where high chemical resistance, low coefficient of friction and sufficient adhesive strength are required at the same time.

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ADDITIONAL INFORMATION

The authors declare that the preparation of the article did not use technologies of generative artificial intelligence and technologies based on artificial intelligence.

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CONTRIBUTION OF THE AUTHORS

Oleg R. Latypov – scientific supervision; analysis of the research results; writing the article; drawing up final conclusions.

Karina V. Kim – editing and data processing.

Vasily V. Lukyanov – scientific supervision; development of the concept and methodology of the research.

Dina R. Latypova – scientific supervision, scientific editing of the text, participation in scientific discussion.

Rustem R. Daminev – scientific supervision, scientific editing of the text, participation in scientific discussion.

Alisa F. Ahmadullina – editing and data processing.

The authors declare no conflict of interest.

The article was submitted 04.07.2026; approved after reviewing 02.10.2026; accepted for publication 08.10.2026.