UDC 620.197.3
DOI: 10.15587/1729-4061.2016.79559

# RESEARCH OF THE MECHANISM OF FORMATION AND PROPERTIES OF TRIPOLYPHOSPHATE COATING ON THE STEEL BASIS

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***Досліджений механізм формування, захисні властивості, мікроструктура, фазовий склад триполіфосфатних покриттів на сталі. Покриття наносили в потенциодинамічному та потенціостатичному режимах в водному розчині натрію триполіфосфату. Запропонований електрохімічний ступінчастий механізм формування триполіфосфатного покриття. Встановлено, що покриття з високими захисними властивостями формується в умовах повної пасивації при потенціодинамічному режимі та складається з двох шарів: перший – тонкий, щільний шар, що містить кристалічну фазу фосфатної природи, і другий – товстий гідрофільний шар, здатний до висихання***

***Ключові слова: натрію триполіфосфат, анодна крива, пасиваційне покриття, захисні властивості, механізм формування***

***Исследован механизм формирования, защитные свойств, микроструктура, фазовый состав триполифосфатных покрытий на стали. Покрытия наносили в потенциодинамическом и потенциостатическом режимах в водном растворе триполифосфата натрия. Предложен электрохимический ступенчатый механизм формирования триполифосфатного покрытия. Установлено, что покрытие с высокими защитными свойствами формируется в условиях полной пассивации при потенциодинамическом режиме и состоит из двух слоев: первый – тонкий, плотный слой, содержащий кристаллическую фазу фосфатной природы, и второй – толстый гидрофильный слой, способный к высыханию***

***Ключевые слова: триполифосфат натрия, анодная кривая, пассивационное покрытие, защитные свойства, механизм формирования***

## 1. Introduction

Research results presented below are aimed at the development of effective and relatively cheap corrosion protection methods of rolled metal goods that would improve ecological safety and economic efficiency of metal rolling industry.

During the manufacturing process, the corrosion protection of metal goods is achieved with the use of an aqueous solution of contact inhibitors of atmospheric corrosion that form a passivating layer on the metal surface. Nitrites, chromates, silicate and phosphates are amongst the most common water-soluble inhibitors. Pipe plants use soda-nitrite, trisodiumphosphate and chromium-containing solutions [1, 2]. While nitrite- and chromate-based solutions produce the most effective conversion coating, it's desired to avoid their use [3, 4], due to their cancerogenic, mutagenic and teratogenic properties. These properties complicate their storage and application. The disposal of spent solutions is also a big concern. The presence of toxic compounds requires special equipment and methods for proper neutralization of scouring waters. If not properly treated, such effluents cause significant contamination of the region waters. In addition, for metals with oxide coats, the pressure treatment, painting and enameling are not possible [1, 2]. Thus renounce of toxic material and search for accessible, cheap and effective methods of conversion coating is a relevant problem.

## 2. Literature review and problem statement

As of late, the polyphosphates find more and more application in metallurgy. Owing to their low toxicity and ability to inhibit steel corrosion and limescale formation, even at low concentrations, they are used in municipal water transfer, and for the protection of thermal and power equipment from corrosion. [1, 5, 6]. Polyphosphates also find their application as additives during cleaning, degreasing and passivation of metal [1, 2], and also in zinc – phosphate solutions for achieving small fine crystalline coats [6, 7], improvement of their protective properties [8, 9] and as anticorrosion paint pigments [10–12]. In addition, steel treatment using polyphosphates doesn't interfere with application of enamel, oil, paint and other materials [1, 2].

Among polyphosphates sodium tripolyphosphate (STPP) is widely used in industry, owing to its non-toxicity and good solubility. Aqueous STPP solution is stable, non-volatile, cheap and available for industrial use. Based on these properties its use in conversion coating of steel is considered promising.

It is known, that mechanism of phosphate and polyphosphate protection implies a potential shift of the protected metal to more positive values via formation of passivating films of variable composition [13–18] and few micrometers thick [1, 6]. No data have been found in the literature regarding the use of STPP as a phosphate-based electrolyte for anticorrosion coat formation nor any information regarding coat structure and properties. Authors of works [5, 6] mainly review STPP as a corrosion inhibitor of iron in neutral media. While in some cases it has been studied in a mixture with dicyclohexylamine nitrite for inhibition of pitting corrosion of 08X13 steel during equipment steaming [19] and chloride corrosion of low-carbon steel [13]. The results obtained in work [13] prove the possibility of complex formation between $Fe^{+2}$ cation and STPP in 1 M HCl solution.

Research results [20–22] have proven the presence of films on the surface of low-carbon steel that have formed in STPP based aqueous solutions of variable composition. Tripolyphosphate [20] and modified tripolyphosphate coats [21, 22] are thin, transparent, and non-porous with high adhesion to the steel substrate and protective properties under conditions of atmospheric corrosion.

However, lack of data on the formation mechanism of such coats and their influence on the anodic dissolution of iron in an aqueous STPP solution, complicates application process and estimation of properties. This limits the effective use of aqueous STPP solution as a non-toxic corrosion inhibitor for rolled metal.

## 3. Aim and objectives

The aim of the work was to determine the formation mechanism of coats on the steel substrate in an aqueous STPP solution.

Objectives:

– to study the corrosion behavior of cold-rolled low-carbon steel in an aqueous STPP solution by using the method of anodic polarization curves;

– to electrochemically deposit coats onto the surface of steel samples in an aqueous STPP solution, under potentiostatic and potentiodynamic conditions, using determined potential values of various passivation plateaus;

– to study protective properties, structure, phase composition of obtained coats by using electrochemical, corrosion, metallographic methods and XRD;

– to conduct a comparative analysis of sample characteristics and to determine formation conditions of the coat with good protective characteristics;

– to assess the mechanism of tripolyphosphate coat formation.

## 4. Materials and methods

Corrosion behavior of low-carbon cold-rolled steel was studied be means of potentiodynamic anodic polarization in a 12 % STPP aqueous solution (pH=8.79) as an electrolyte, at 20–22 °C. Two sets of electrode samples (05kp steel) were used in the study. Samples from the first set were of circular shape with a thickness of 0.3 mm. Samples from the second set were square-shaped with a thickness of 1 mm. The chemical composition of both sample sets was the same. Up to 20 samples of each set were used in experiments. The experiments were duplicated 2–3 times.

In order to study the formation mechanism of tripolyphosphate coats on the steel surface, the coats were deposited electrochemically by potentiostatic and potentiodynamic methods. The deposition potentials were chosen based on results of potentiodynamic anodic polarization.

On the first set of samples, the coat was deposited potentiodynamically by applying an anodic linear sweep (sweep rate 1mV/s) from starting potential to a potential value of corresponding passivation plateau $E_0$=+83 mV; $E_1$=+390 mV; $E_2$=+917 mV; (vs NHE).

Protective properties of deposited coats were studied by means of potentiodynamic anodic polarization in a 0.1 N aqueous $Na_2SO_4$ solution to model atmospheric corrosion [1, 2].

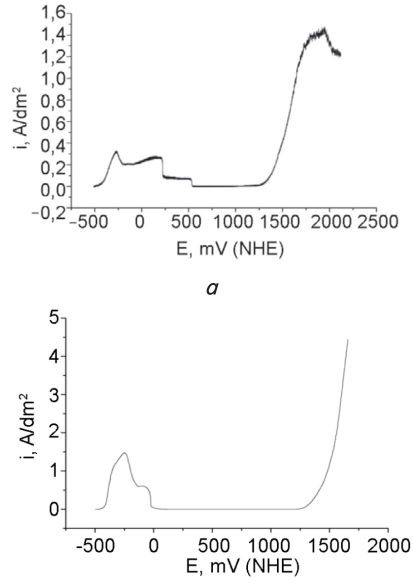
On the second set of samples, coats were deposited potentiostatically and potentiodynamically. For potentiostatic deposition, a potential value, corresponding to a value of passivation plateau, was applied to a working electrode: $E_1$=–87 mV or $E_2$=+619 mV (vs NHE). The potentiodynamic treatment was carried out by an anodic linear sweep (sweep rate 1 mV/s) from stationary potential to potential values of corresponding passivation plateau mentioned above.

XRD patterns were recorded on DRON-3 X-ray diffractometer (USSR), Cu–Kα (λ=1.54178 Å).

The microstructure of initial surface of samples and deposited coats was studied using Scanning Electron Microscope 106-I (SELMI, Ukraine) and optical microscope GX-51 (Olympus, Japan).

## 5. Experimental results

Fig. 1 shows anodic polarization curves of 05 kp steel electrodes in an aqueous STPP solution.



*a*

*b*

Fig. 1. Anodic polarization curves of 05kp steel electrodes in an aqueous STPP solution: *a* — first set; *b* — second set

The anodic polarization curve of the first sample set in an aqueous STPP solution (Fig. 1, *a*) demonstrates three passivation plateaus. A "null" passivation plateau can be observed in the potential range from –210 to +220 mV. The anodic dissolution current density on this plateau rises and is about 0.2 A/dm$^2$. Afterwards, a sharp drop in anodic current density to 0.07 A/dm$^2$ is observed, which corresponds to the first passivation plateau. In the potential range from +530 to +1250 mV, the second passivation plateau with the current density of zero is observed. Metal dissolution with oxygen evolution occurs at potential values above +1250 mV (Fig. 1, *a*).

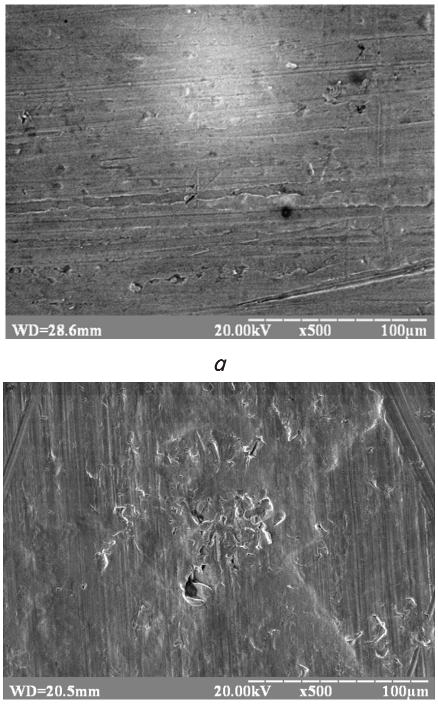
The anodic polarization curve of the first sample set in an aqueous STPP solution (Fig. 1, *b*) demonstrates partial and complete passivation plateaus. "Null" passivation plateau cannot be observed (Fig. 1, *b*). Partial passivation plateau is situated in the potential range from –125 to +0.00 mV, with anodic dissolution current density rapidly dropping from 0.50 A/dm$^2$ to 0.00 A/dm$^2$ (Fig. 1, *b*). In the potential range from +0.00 to +1250 mV, complete passivation plateau is observed.

Fig. 2 shows SEM surface images of 05 kp steel sample at 500 times magnification. The images demonstrate microstructure inhomogeneity of initial surface of the second set samples.

Fig. 3 shows anodic polarization curves in 0.1N $Na_2SO_4$ for 05 kp steel and steel coated potentiodynamically in an aqueous STPP solution to potential values of various passivation plateaus.

The anodic curve for bare steel (Fig. 3, *a*) demonstrates a plateau within the potential range +250 to +1750 mV with current density of 1.5 A/dm$^2$, that corresponds to decrease of the metal dissolution rate.

For samples coated at potentials +83 mV ("null" passivation plateau) and +390 mV (first passivation plateau) (Fig. 3, *b, c*), anodic curves in positive potential values demonstrate partial passivation plateaus. The current densities are 1.25 and 1.00 A/dm$^2$, correspondingly.

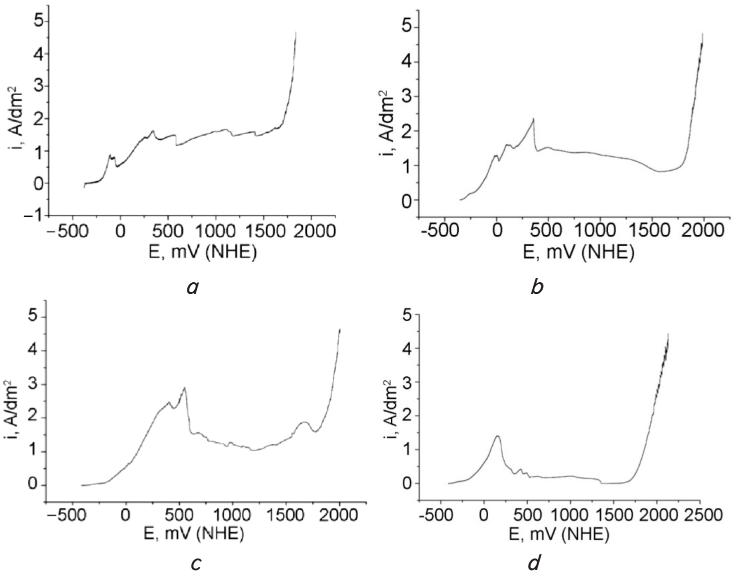


*a*

*b*

Fig. 2. SEM images of low-carbon cold-rolled steel (05 kp): *a* — first set; *b* — second set, Magnification — ×500

The anodic curve for coats deposited under conditions of complete passivation at E =+920 mV (second passivation plateau), is shown in Fig. 3, *d*.



*a*

*b*

*c*

*d*

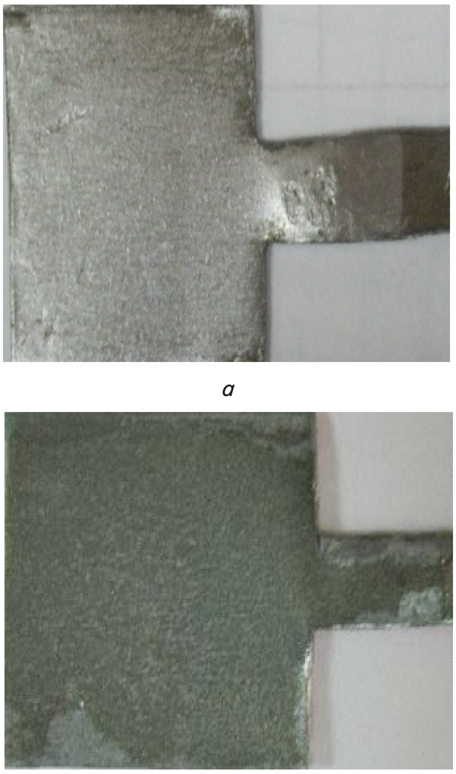
Fig. 3. Anodic polarization curves in 0.1N $Na_2SO_4$ for 05 kp steel and steel coated potentiodynamically in an aqueous STPP solution to potential values of various passivation plateaus:
*a* — bare 05kp; *b* — 05 kp coated (E=+83 mV);
*c* — 05 kp coated (E=+390 mV); *d* — 05 kp coated (E=+920 mV)

In the potential range of +500 to +1500 mV, a significant decrease of current density from 0.20 A/dm$^2$ to 0.00 A/dm$^2$ can be observed – the complete passivation plateau.

After potentiodynamic deposition, a relatively thick, gel-like, semi-transparent, gray-brown film is formed on the samples surface (Fig. 4, *b*). After washing and film removal, the metal surface looks white and shiny (Fig. 4, *a*).

In Fig. 5 cracks in the dried film can be observed.

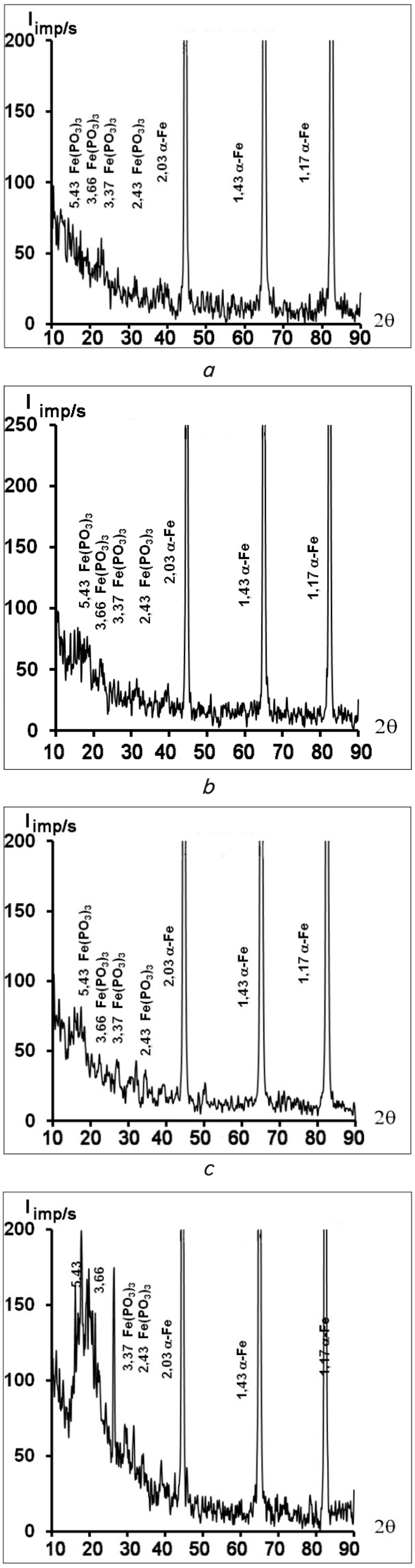
X-ray amorphous phase of phosphate nature can be observed on XRD patterns for coats deposited potentiostatically (Fig. 6, *a, b*). XRD patterns for coats deposited potentiodynamically show the presence of X-ray amorphous phase (Fig. 6, *c*) and crystalline phase (Fig. 6, *d*) of phosphate nature.


*a*

*b*

Fig. 4. Samples of 05 kp steel coated potentiodynamically in an aqueous solution of STPP:
*a* – after washing; *b* – before washing


Fig. 5. Film potentiodynamically deposited on the 05 kp steel sample in an aqueous STPP solution at the potential of the first passivation plateau; ×1500 magnification. Red circles highlight cracks in the films



*a*

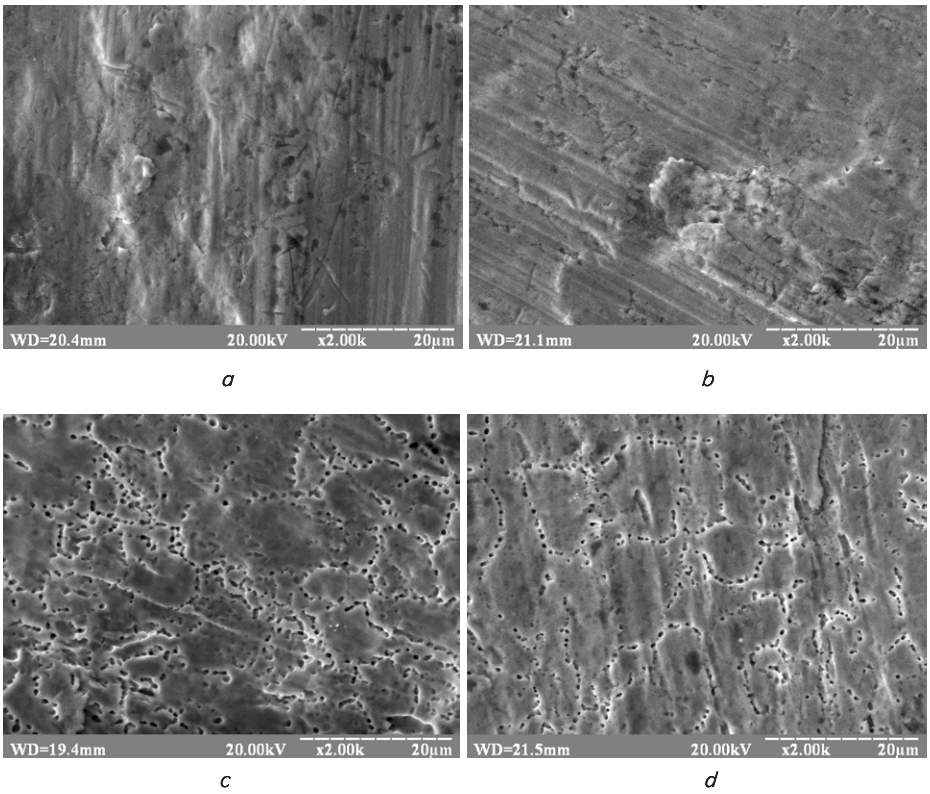
*b*

*c*

*d*

Fig. 6. XRD patterns of 05 kp coated in an aqueous STPP solution by different methods and under different conditions:
*a* – potentiostatic, first passivation plateau;
*b* – potentiostatic, second passivation plateau;
*c* – potentiodynamic, first passivation plateau;
*d* – potentiodynamic, second passivation plateau

Fig. 7, *a, b* show~~s~~ the structure of potentiostatically deposited thin coats. Chain fragments are present in a structure of thicker films that were deposited potentiodynamically (Fig. 7, *c, d*).



*a* *b*

*c* *d*

Fig. 7. SEM images of 05 kp coated in an aqueous STPP solution by different methods and under different conditions: *a* — potentiostatic, first passivation plateau; *b* — potentiostatic, second passivation plateau; *c* — potentiodynamic, first passivation plateau; *d* — potentiodynamic, first passivation plateau; ×2000 magnification

## 6. Discussion of research results of anodic behavior of low-carbon cold-rolled steel in aqueous STPP solution, structure and properties of formed coats

It is known, that the decrease of the metal dissolution rate during anodic polarization is related to the formation of passivation layers of various natures on the metal surface [1, 5, 6, 13–15].

Thus, in our case, starting from "null" passivation plateau, a protective film is formed on the metal surface that is further modified two times.

During transition from one passivation plateau to another, a decrease of current density to zero is observed, which indicates the composition, properties change or film compacting. This allows to assume that film formation on the metal surface in STPP solution occurs in a few stages. During "null" passivation plateau, with the average potential of +83 mV, the I-st stage is realized. During this stage, a film of adsorptive nature with weak protective properties is formed, which is indicated by a further increase of current density. The presence of such film doesn't interfere with metal dissolution and formation of $Fe^{2+}$ ions. Afterwards, in the boundaries of the "first" passivation plateau, the II-nd stage is realized. Probably, under these conditions the film is formed as a result of a reaction between $Fe^{2+}$ cations and tripolyphosphate anion. The current densities and potential values, corresponding to this region of the anodic polarization curve, indicate partial metal passivation, meaning that the film has poor protective properties. Further potential shift into the anodic region is accompanied by the appearance of a broad complete passivation plateau. Presumably, in this case the stage III is realized – formation of a film with good protective properties, owing to self-compacting. During this, possibly, the oxidation of $Fe^{2+}$ to $Fe^{3+}$ occurs, resulting in film composition change.

The anodic polarization curve for 05 kp steel second set (Fig. 1, *b*) demonstrates partial and complete passivation plateaus. In addition, the "null" passivation plateau~~s~~ are almost non-existent, current densities at the plateau boundaries are higher, and potential values are more negative than for samples from the first set.

Such difference in anodic behavior between two sets of steel of the same grade can be related to the difference in chemical reactivity.

From Fig. 2 it can be seen that the microstructure of samples from the second set of 05 kp steel is characterized by a higher degree of inhomogeneity.

From the perspective of electrochemistry, the more inhomogeneous surface can be considered more reactive. It is possible, that on such surface the first stage – the formation of adsorbed film occurs rapidly, and the stage is realized before recording of the polarization curve. As a result, the "null" passivation plateau is absent on the anodic curve for samples from the second set, but has different features, listed above (Fig. 1, *b*).

Thus, the realization of assumed multi-stage formation mechanism of tripolyphosphate coat on steel assumes a few passivating coats with different composition and properties exist in this system. The study on the properties and structure of such coats would allow to obtain additional data during determination of the formation mechanism.

In order to evaluate protective properties of coats deposited on 05 kp steel, the polarization curves were recorded in 0.1 N $Na_2SO_4$ solution, in order to model atmospheric corrosion [1, 2]. Samples from the first set with coats deposited potentiodynamically in an aqueous STPP solution, to potential values of various passivation plateaus: «null» (E=+83 mV); first (E=+390 mV); second (E=+920 mV).

Analysis of the anodic curve for bare steel (Fig. 3, *a*) showed, that the plateau corresponding to a decrease of the metal dissolution rate, is situated in the potential range from +250 to +1750 mV with approximate current density of 1.5 A/dm$^2$. Probably, in this case, the current density is limited by the removal rate of corrosion products or by the formation of the unstable salt-oxide film. It is unlikely that such film can protect metal from further corrosion. Thus the passivation current density increases with increasing potential, while it is decreasing for coated samples.

Thus, the deposited coats offer different degrees of corrosion protection (Fig. 3, *a–d*). For instance, the protective properties of the film deposited at E=+390 mV (first passivation plateau), are also negligible. However, it can be considered, that they are somewhat better that those of bare samples and coated ("null" plateau), because limiting current density with this coat is about 1.00 A/dm$^2$ (Fig. 3, *c*). Only the coat deposited under conditions of complete passivation at E =+920 mV (second passivation plateau), is assumed to have good protective properties. The anodic curve for this film in the potential range from +500 to +1500 mV demonstrates complete passivation plateau, with a decrease of current density from 0.20 A/dm$^2$ to 0 A/dm$^2$.

The analysis of this data has shown that protective properties of the studied coats are different. Additionally, the continuous increase of protective properties during the potential sweep from starting potential to "null" and last passivation plateaus, can be considered as a proof of assumed multi-stage formation mechanism of coat on steel in an aqueous STPP solution.

Based on visual observation, during potentiostatic anodic treatment of samples from the second set at potential values of passivation plateaus occurred rather slowly, and the current density was almost zero, meaning the electrochemical process almost did not happen. Visual examination after treatment and washing did not show any color change of the sample surface in comparison to the initial state. When treatment method was changed to potentiodynamic, the nature of formation process changes significantly. Under these conditions, a relatively thick, gel-like, semi-transparent, gray-brown film is formed on the samples surface. If necessary, the freshly deposited film can be easily removed (Fig. 4, *a*). After film removal, the metal surface looks white and shiny (Fig. 4, *a*). These facts prove that during potentiodynamic deposition the films are formed due to the anodic dissolution of iron. The metal surface has a similar look after electrochemical polishing which occurs with film formation from dissolution products. Upon drying of the gel-like thick film, its thickness is decreased while adhesion to the metal surface increases drastically. It is not possible to remove dried film. Additionally, cracks appear on the coat surface (Fig. 5). It can be concluded that during potentiodynamic deposition a film of matrix structure is formed, that is composed of two layers: the first – thin compact layer, the second – thick hydrophilic layer.

In order to prove the existence of the first layer, samples with coats deposited potentiostatically and potentiodynamically, were immediately washed, and the outer thick layer was removed. Afterwards, the XRD patterns of samples were recorded. From XRD patterns (Fig. 6, *a, b*) it can be seen that the structure of potentiostatically deposited films is X-ray amorphous. The same structure (Fig. 6, *c*) can also be observed for the film deposited potentiodynamically to the potential of the first passivation plateau. Reflexes of the crystalline phase of phosphate nature can only be observed in the XRD pattern for the film deposited potentiodynamically to the potential of the complete passivation plateau (Fig. 6, *d*)

Microstructural analysis of the coats formed on the steel surface under various treatment regimes in an aqueous STPP solution; has shown significant structural differences of deposited coats. As can be seen from Fig. 7, *a, b*, the potentiostatic treatment leads to formation of thin coats. The topography of these films is almost indifferent (Fig. 7, *a, b*) topography of the initial steel substrate (Fig. 2). For the films deposited potentiodynamically (Fig. 7, *c, d*) from stationary to potentials of passivation plateaus, the microstructure changes. The coats look thicker, and the underlaying metal substrate cannot be seen. Chain fragments can be observed in the coat structure. Their appearance is probably related to the electrochemical dissolution of the metal substrate. In place of significant inhomogeneity, this process occurs at a greater rate leading to formation of chain fragments.

The acquired results prove the multi-stage electrochemical mechanism of tripolyphosphate coat formation and the necessity of each stage to occur involving intermediate products.

The results of conducted complex studies have allowed to determine the nature of electrochemical multi-stage formation mechanism of tripolyphosphate coats and features of their structural and phase composition. The knowledge acquired is a theoretical and practical basis of further studies that can be directed at the development of effective deposition methods of tripolyphosphate coats with estimated set of properties for the protection of metal goods from atmospheric and high-temperature gas corrosion.

## 7. Conclussions

1. The anodic behavior of cold-rolled low-carbon steel in an aqueous STPP solution at room temperature has been studied. Up to three passivation plateaus can be observed on the potentiodynamic curves that characterize the anodic behavior of 05 kp steel. Regardless of the metal surface activity, the anodic sweep ends with complete passivation plateau with the anodic current density equal to zero.

2. The electrochemical formation mechanism of passivation coat formation in an aqueous STPP solution on the surface of 05kp steel has been proposed. The mechanism is realized in stages: at the beginning an adsorption film is formed on the metal which afterwards is modified two times accompanied by a change of properties and composition. The mechanism includes three stages:

– I – start of the passivation and formation of adsorptive film with poor protective properties;

– II – partial passivation: formation of film with weak protective capabilities as a result of reaction of tripolyphosphate anion with Fe$^{2+}$ions;

– III – complete passivation and formation of the most protective film, because of self-compacting and, probably, oxidation of Fe$^{2+}$ ions to Fe $^{3+}$.

3. The rate of first adsorption stage is likely related to the activity of metals surface. The higher the surface activity the faster the adsorption stage occurs.

4. It has been established that coat with good protective properties only forms under conditions of complete passivation in potentiodynamic regime. This proves the multi-stage electrochemical mechanism and necessity of each stage to occur involving intermediate product to form a coat with best protective properties

5. It has been established that coat formed in an aqueous STPP solution by potentiodynamic method under conditions of complete passivation, is composed of two layers: first – thin, compact layer, that contains a crystalline phase of phosphate nature, and second – thick hydrophilic layer, capable of drying.

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