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STUDY OF THE ANTICORROSION EFFECT OF POLYMER PHOSPHATES ON STEEL AT ELEVATED TEMPERATURES

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Встановлено, що в області температур гарячої деформації сталі розплави поліфосфатів розчиняють оксиди заліза, що містяться в окалині. Метафосфат натрію взаємодіє з оксидом заліза з утворенням змішаних поліфосфатів. Отримані продукти в чотири рази збільшують час до появи перших проявів корозії та до сорока разів зменшують ступінь корозійного ураження сталі. Це обумовлено блокуванням поверхні плівкою змішаних поліфосфатів та забезпеченням значень рН близько 9 в атмосферних умовах

Ключові слова: метафосфат натрію, триполіфосфат натрію, змішані поліфосфати, протикорозійна дія, окалина

Установлено, что в области температур горячей деформации стали расплавы полифосфатов растворяют окислы железа, содержащиеся в окалине. Метафосфат натрия взаимодействует с оксидом железа с образованием смешанных полифосфатов. Полученные продукты в четыре раза повышают время до появления первых следов коррозии и до сорока раз уменьшают степень коррозионного поражения стали. Это обусловлено блокировкой поверхности пленкой смешанных полифосфатов и обеспечением значений рН около 9 в атмосферных условиях

Ключевые слова: метафосфат натрия, триполифосфат натрия, смешанные полифосфаты, противокоррозионное действие, окалина

1. Introduction

Conversion treatment of steel products is a common type of surface treatment. This is predetermined by the need to protect corrosive-active steel surface against aggressive effect of the environment. Phosphate coating is typically applied as a conversion coating. Such a coating can act as the adhesion layer for paint and varnish treatment, oil-retaining or a separate protective film.

Solutions based on phosphates and polyphosphates are ecologically tolerant, they are affordable and easy to use.

Despite the long history of employing such solutions in machine building, the scope of applications of phosphate

treatment is constantly expanding. One of the directions of active use of phosphate blends in modern manufacturing is the modification of tribological characteristics of the surface of steel products. In this case, polyphosphates act as an inorganic grease for the steel surface treated at elevated temperature. It should be noted that the high-temperature treatment of steel is accompanied by the oxidation of surface layers with the creation of dross and pockets of corrosion destruction.

Given this, there is a relevant, technologically and economically motivated, task on the optimization of anti-frictional polyphosphate compositions that would ensure high anti-corrosion properties of the steel surface under conditions of high-temperature treatment.

2. Literature review and problem statement

Phosphate anticorrosive treatment is used in many areas of production [1]. Papers [2, 3] address effect of the nature of a steel sample on thickness and protective properties of phosphate coatings for the surface of reinforcement of ferro-concrete structures. Authors of [4] investigated influence of temperature and concentration of NaNO_2 in a phosphating bath on the surface morphology of iron-phosphate coatings of low-carbon steel. It is shown that an increase in the temperature of phosphating bath that does not contain NaNO_2 to 70°C contributes the increase in the continuity of phosphate coatings. Introduction of NaNO_2 to the solution leads to substantial improvement in the continuity of coatings. Phosphate layers were applied on the soft magnetic materials Fe and FeSiAl as insulating coatings [5]. It was established that the surface passivation of iron with a solution of phosphoric acid results in the formation of amorphous iron phosphate. The coating formed on FeSiAl mainly consists of $\text{Al}(\text{PO}_3)_3$. After annealing at high temperatures (600°C), iron phosphate decomposes into phosphides and iron oxides. A protective layer of $\text{Al}(\text{PO}_3)_3$ transforms into Al_2O_3 and P_2O_5 . In order to improve protective characteristics of zinc-phosphate conversion coatings on the high-strength steel, it was proposed to perform phosphate treatment under ultrasonic treatment of the working solution [6]. Improvement in the corrosion resistance of zinc-phosphate coatings on the soft steel [7] was achieved by introducing zinc oxide nanoparticles to the phosphating solution.

Comparison of the results of examining magnesium-phosphate and zinc-phosphate coatings on the low-carbon steel showed [8] that the thickness and protective ability of magnesium-phosphate films was three times higher. Magnesium phosphate coatings, cathodically formed on the low-carbon steel, also demonstrated high anticorrosion properties [9]. This is due to the formation of the Newberyite phase ($\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$) on the steel surface. An increase in the temperature of phosphating leads to the increase in thickness and protective properties of magnesium-phosphate films [10]. Magnesium-phosphate coatings in papers [11, 12] were applied on the high-alloyed steel.

Authors of [13, 14] proved that phosphating can be considered an effective way to improve the biocompatibility of iron applied in biomedicine.

A number of techniques were proposed to increase the anti-corrosive properties of phosphate coatings. Thus, an increase in the protective ability of a conversion coating in [15] is achieved by introducing rare earth elements to the solution of phosphating. Phosphate coatings with improved protective characteristics are also received from solutions containing polyvinyl alcohol [16]. Zinc-phosphate composites with SiO_2 nanoparticles [17] demonstrated higher protective properties compared to those in conventional coatings. Introduction of polyphosphates to zinc-phosphate solutions leads to the formation of microcrystalline conversion coatings on steel with enhanced protective properties [18].

In addition, phosphates and polyphosphates of zinc and aluminum are used as corrosion pigments in epoxy coatings [19, 20].

Quite common is the application of polyphosphates for rendering anti-frictional properties to steel surface [21]. In [22], authors investigated tribochemical properties were of amorphous layers of zinc polyphosphate with a different length of the polymer chain. It was found that the tribological load did not change the composition of short-chain

polyphosphates. Long-chain polyphosphates are prone to depolymerization.

When machining steel at high temperatures, polyphosphates pass in the molten state [23, 24]. A melt of polyphosphate reduces the coefficient of friction of metal surfaces. In the molten state polyphosphates interact with the steel surface, which leads to the high adhesion of film. Tribological study at 800°C showed [25] that sodium metaphosphate reduces coefficient of friction by 49%. It is assumed that this is due to the formation of short-chain amorphous compounds of the types NaFePO_4 and $\text{Na}_3\text{Fe}(\text{PO}_4)_2$.

Thus, the use of polyphosphates during high-temperature machining of steel products as a technological grease appears very promising. At the same time, steel formation at high temperature is a complex process, which is accompanied by thermal, microstructural, physical and chemical transformations in the surface layer of articles [26]. The attention of researchers is concentrated in these systems mainly on the tribological characteristics of polyphosphates [23–25]. However, the ability of polyphosphates to protect steel surface from corrosion requires a comprehensive approach when choosing components of technological greasing. Antifriction polyphosphate compositions should enable maximum protection of the treated steel surface against corrosion. In this regard, it is very important to establish the anti-corrosive effect of polyphosphates on steel at elevated temperatures.

3. The aim and objectives of the study

The aim of present work is to establish the anti-corrosive effect of polyphosphates on steel at elevated temperatures.

To achieve the set aim, the following tasks have been solved:

- to investigate effect of temperature on the thermal stability of metaphosphate and sodium tripolyphosphate and define products of interaction between with polyphosphates and scale;
- to determine the nature of protective action of a technological lubricant based on metaphosphate and sodium tripolyphosphate and to compare the extent of corrosion damage to steel treated in the presence of polyphosphate lubricant and sodium chloride.

4. Materials and methods for examining anti-corrosive effect of polyphosphates on steel

Metaphosphate used in the present work was synthesized from sodium orthophosphate. Sodium metaphosphate (NaPO_3)_n was received by heating crystalline hydrate of sodium orthophosphate mono-substituted in ceramic cuvettes. Heating temperature was to 700°C . Next, the resulting melt was cooled on stainless steel. The synthesized sodium metaphosphate was crushed to the powder-like state. Reagent was stored in a desiccator over sulphuric acid. Composition of the obtained powder was determined by the method of eluent ion exchange chromatography [27]. Chromatographic separation of phosphates was performed in a glass column with a diameter of 15 mm and a length of 90 mm. The ion-exchange resin was the anionite of brand IF-23 (Ukraine). Determining mass fraction of P_2O_5 in samples was conducted according to GOST 20291-80, using the photoelectrocolorimeter FEC-56M (Russia) at a wavelength of 440 nm.

We used technical sodium tripolyphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$) during study.

For the model studies, we used scale that was received at a temperature of 800 °C in an annular gas furnace. Granulometric composition of the scale was normalized using a sieve with holes of diameter 50 μm. We used powder with particle size less than 50 μm.

To register thermograms, we applied the derivatograph Q-1500 D (Hungary), the Paulik-Paulik-Erdey system. Derivatographic study was conducted in atmospheric air in the temperature range 20–1000 °C with a heating rate of samples equal to 10 K/min.

Determining the composition of products of interaction between scale and the melt of metaphosphate and sodium tripolyphosphate was carried out using an X-ray phase analysis. Model mixture consisting of metaphosphate or sodium tripolyphosphate (80 % by weight) and scale (20 % by weight of Fe₂O₃) was calcined in a muffle furnace at 800 °C for 20 minutes. The cooled glass-like mass of black color was crushed and analyzed.

Radiographs were received using the diffractometer DRON 3.0 (Russia) under CuK_α -radiation. Interplanar distances on radiographs of the samples were compared to data from catalogues of ASTM (American Society for Testing and Materials).

Surface morphology of steel samples was studied using the raster electron microscope REMMA 102-02 (Ukraine).

5. Results of research into interaction between polyphosphates and scale

Metaphosphate and sodium tripolyphosphate are key components of the antifriction composition [28] that acts as a technological lubricant during hot rolling of steel seamless pipes. In order to identify the nature of the anti-corrosive effect of polyphosphates on steel, it is required to identify the processes that occur at operating temperature. The value of a given temperature reaches 1000 °C. Taking into consideration that under such circumstances the steel surface is covered with a layer of scale, we shall determine phase composition of the latter. Radiographs (Fig. 1) shows that the examined sample represents mainly a mixture of hematite (α-Fe₂O₃), maghemite (γ-Fe₂O₃), and the impurity FeO. Obviously, at elevated temperatures, the examined polyphosphates are able to interact with these oxides.

By employing a derivatographic study, it is possible to follow thermochemical transformations taking place in the system polyphosphate-scale. Thermogram of the model system consisting of 80 % by weight of scale and 20 % by weight of sodium metaphosphate is shown in Fig. 2.

The first exothermic effect along a DTA curve corresponds to the peak at a temperature equal to 130 °C. Obviously, there is moisture desorption. It should be noted that the surface of a finely-dispersed phase of polymer phosphates carries a hydroxide layer. Hydroxides act as centers of sorption of molecular water, which is trapped on the surface due to the formation of hydrogen bonds.

Endoeffects, observed at temperatures of 250 °C and 380 °C, should be attributed to the dissolution of Fe₂O₃ in the crystals of sodium metaphosphate. The endoeffect, which corresponds to a temperature of 610 °C, is predetermined by the melting of sodium metaphosphate. In this case, a phase transformation occurs with the formation of mixed triphosphate Na₉Fe₂(P₃O₁₀)₃.

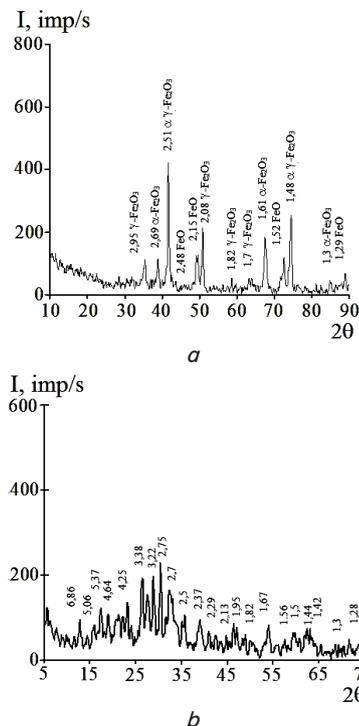


Fig. 1. Radiographs: a – scale; b – products of interaction between scale and sodium metaphosphate

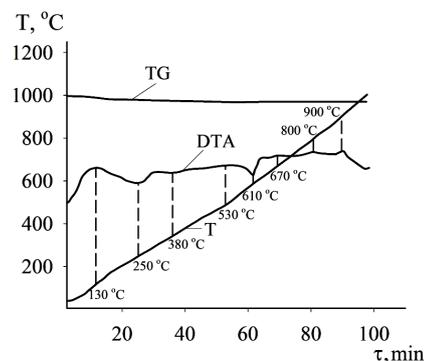
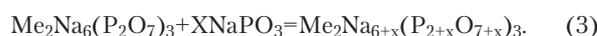


Fig. 2. Thermogram of the model system consisting of 80 % by weight of scale and 20 % by weight of sodium metaphosphate

Exoeffects at temperatures of 670 °C, 800 °C and 900 °C should be treated based on the following considerations. First, there is a dissolution of scale in molten sodium metaphosphate. Next, a chemical interaction between Fe₂O₃ and sodium metaphosphate leads to the separation of mixed polyphosphates with a higher melting temperature. When temperature reaches 900 °C, there occurs the melting of mixed polyphosphates.

Interaction between the oxide of a trivalent metal and NaPO₃ can be represented by the following reactions [28]:



Radiograph of products of interaction between scale and sodium metaphosphate (Fig. 1, b) shows no peaks corre-

sponding to α -Fe₂O₃ and γ -Fe₂O₃. In addition, the maxima at d_{HKL} =5.70; 3.0; 3.04 Å, characteristic of sodium metaphosphate, are also absent. Therefore, as a result of the thermochemical transformations, almost all of the scale and sodium metaphosphate participated in reactions (1)–(3) with the formation of mixed polymer phosphates of sodium and iron.

Thermogram of the model system consisting of 80 % by weight of scale and 20 % by weight of sodium tripolyphosphate is shown in Fig. 3. In the region of low temperatures, there are two peaks observed. Exoeffects at temperatures of 100 °C and 160 °C are due to the desorption of physically sorbed moisture.

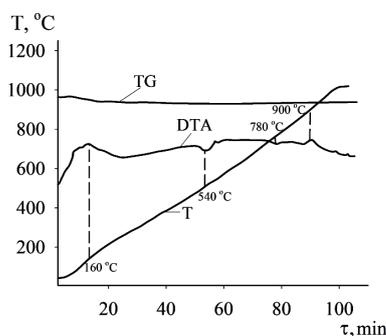


Fig. 3. Thermogram of the model system consisting of 80 % by weight of scale and 20 % by weight of sodium tripolyphosphate

Note that anhydrous sodium tripolyphosphate that contains no impurities is thermally stable until the melting temperature. Destruction of sodium tripolyphosphate at temperatures lower than the melting temperature is possible in the case of presence of foreign substances in the system. Endoeffects, observed at temperatures of 540 °C and 580 °C, are probably caused by the insufficient purity of the examined tripolyphosphate. When the temperature rises to 662 °C, there occurs the melting of Na₅P₃O₁₀, accompanied by condensation processes with the formation of high-molecular polyphosphates. A rise in the temperature of the melt to a temperature of 880 °C leads to an increase in the content of tripoly- and tetra polyphosphates [29].

Identification of the products of interaction between scale and sodium tripolyphosphate can be conducted based on an X-ray phase study. Radiograph of the model mixture, calcined at 800 °C, is shown in Fig. 4. Diffraction maxima at d_{HKL} =4.45, 2.76 Å correspond to sodium tripolyphosphate Na₅P₃O₁₀, which probably do not interact with scale.

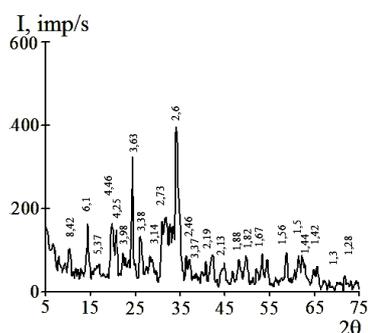


Fig. 4. Radiograph of the products of interaction between scale and sodium tripolyphosphate

Peaks at d_{HKL} =2.60, 3.38, 3.63, 4.25, 6.10 Å can be predetermined by the presence in the mixture of oligophosphates

and the products of interaction between these oligophosphates and scale.

Thus, the more reactive in the interaction with scale is sodium metaphosphate. Mixed polymer phosphates of sodium and iron, formed in the region of high temperatures, improve antifriction properties of the surface of steel.

6. Discussion of results of research into anti-corrosive effect of polyphosphates on steel at elevated temperatures

An anticorrosive effect of polymeric phosphates on steel can be estimated when comparing data on corrosion tests of samples that were exposed to high temperature deformation in the presence of the examined polyphosphates and sodium chloride. Results of field tests are summarized in Table 1. First point traces of corrosion on samples made of steel 20, treated in the presence of metaphosphate and sodium tripolyphosphate, emerged in 30 days. For the samples treated in the presence of sodium chloride, a given indicator was only 7 days. The degree of corrosion damage for the respective samples was 2 % and 74 %. After 60 days of field tests, these values grew to 3.5 % and 100 %, respectively.

Table 1

Results of field tests of steel samples after high-temperature deformation in the presence of polyphosphate lubrication and sodium chloride

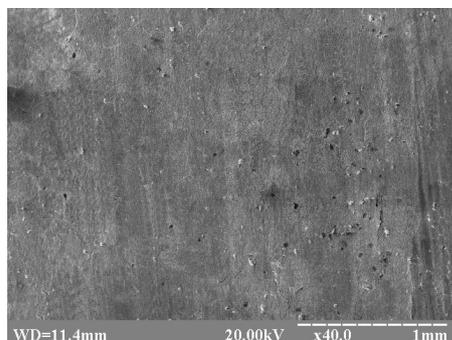
Corrosion test parameters	Technological grease based on	
	sodium chloride	metaphosphate and sodium tripolyphosphate
Duration of sample holding under natural conditions until the emergence of first traces of corrosion, days	7	30
Degree of corrosion damage to steel after 30 days of field tests, %	74	2
Degree of corrosion damage to steel after 60 days of field tests, %	100	3.5

Difference in the degree of corrosion damage to steel surfaces, treated with a polyphosphate lubrication and sodium chloride, is shown in Fig. 5. In the first case, the surface after corrosion tests remained smooth and undamaged, without foreign inclusions (Fig. 5, *a*). When sodium chloride was used as a technological grease, the surface was completely damaged by corrosion (Fig. 5, *b*).

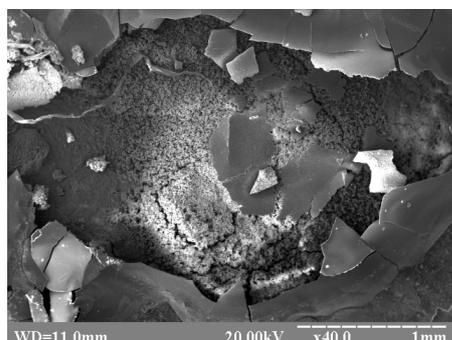
An anticorrosive effect of technological lubricant based on metaphosphate and sodium tripolyphosphate is due to a comprehensive action. High buffer capacity of polyphosphates makes it possible to maintain constant acidity of technological lubrication at the level of pH=8.5–9.5. This contributes to reducing the rate of corrosion. In addition, surface scale interacts with polyphosphates in line with reaction equations:



In this case, mixed polymer phosphates are created that form a protective layer on the surface of steel.



a



b

Fig. 5. Micro images of surface samples after corrosion field tests: *a* – steel 20, treated in the presence of polyphosphate lubrication; *b* – steel 20, treated in the presence of sodium chloride

Thus, the established chemistry of reactions of interaction between scale and the examined polyphosphates

makes it possible to predict the anti-corrosive effect of an anti-friction lubricant on steel. A comprehensive approach to the selection of components of technological lubricants for high-temperature steel treatment, including an assessment of the protective properties, enables optimization of the composition of lubricants in order to improve service resource of steel products. An empirical technique for obtaining the necessary data causes certain labor-intensity in the selection of polyphosphates. It therefore seems promising to establish common patterns in the formation of protective films on steel at high-temperature treatment that depend on the composition and structural characteristics of polyphosphates.

7. Conclusions

1. It is shown that at a temperature of 610 °C there occurs the formation of mixed triphosphate $\text{Na}_9\text{Fe}_2(\text{P}_3\text{O}_{10})_3$. A rise in temperature to 900 °C leads to the formation and melting of mixed polyphosphates. It is shown that scale and sodium metaphosphate interact with the formation of mixed polymeric phosphates of sodium and iron. Results of studying the products of interaction between polyphosphates and scale indicate a greater reactivity of sodium metaphosphate in comparison with sodium tripolyphosphate. It was established that in the region of high temperatures mixed polymer phosphates of sodium and iron, formed at the surface, act as the anti-friction grease.

2. It was established that when using a polyphosphate lubricant, a degree of corrosion damage amounted to 3.5 % of the sample surface. The surface of steel, treated similarly in the presence of sodium chloride, was damaged with corrosion by 100 %. High protective properties of polyphosphate lubricant are due to the formation of a barrier film at the steel surface, consisting of mixed polymer phosphates. In addition, the presence of such a film maintains high pH values under atmospheric conditions.

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