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Investigation of the Corrosion Process by X-Ray Phase Analysis

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Abstract. This article presents an assessment of corrosion processes by X-ray phase analysis and the inhibitory ability of inhibitors using powder diffractometry (X-ray phase analysis) and scanning electron microscopy.

Key words. Goethite, hematite, magnetite, solution, X-ray diffraction analysis, inhibitor.

Corrosion of metals causes serious damage to both industrial equipment and the environment. An economical and effective means of reducing corrosion is the use of inhibitors. At present, a large number of compounds of various nature have been developed that have an inhibitory effect. However, the increasingly complex conditions of production processes require the creation of new highly effective inhibitors [1-8].

The main way to protect against corrosion of metal is the creation of protective coatings - metallic, non-metallic or chemical.

The inhibitory ability of the inhibitor IK-MK-21 was evaluated using a powder diffractometer (X-ray phase analysis) and a scanning electron microscope.

To study the effect of inhibitors on the surfaces of steel grade St.3, 3 iron washers with a diameter of 15 mm were made from a sheet of steel (iron), and one of the washers was lowered into 0.02 wt.% aqueous solution of IK-MK-21 for 24 hours (sample No. 1). The second washer was tested under the action of hydrochloric acid (pH=2) for 24 hours (sample No. 2), the other washer under the action of alkali (pH=11) for 24 hours (sample No. 3). All three washers were dried first in the open air, then in an oven to constant weight. The analysis was carried out on a powder diffractometer and on a scanning electron microscope.

Pictures of the surface areas of samples No. 1, 2 and 3, obtained on a powder diffractometer, are shown in Figures No. 1-3.

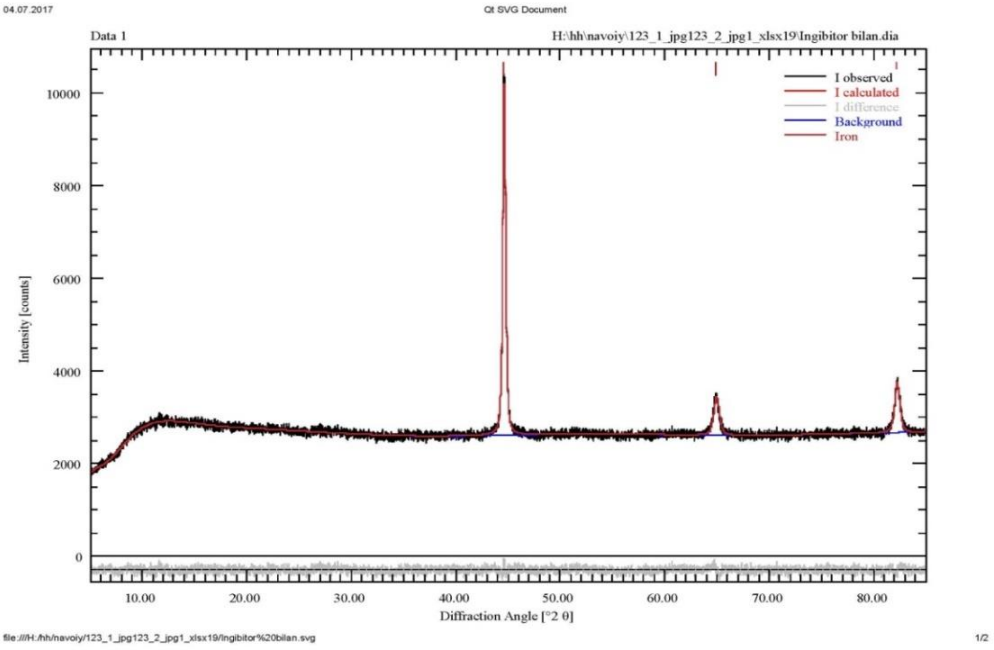

Fig.1. X-ray phase analysis by the Rietveld method on sample No. 1
grade 3 in 0.02 wt. % aqueous solution of IK-MK-21 for 24 hours at a temperature of 20 ^{0}C (pH = 11).

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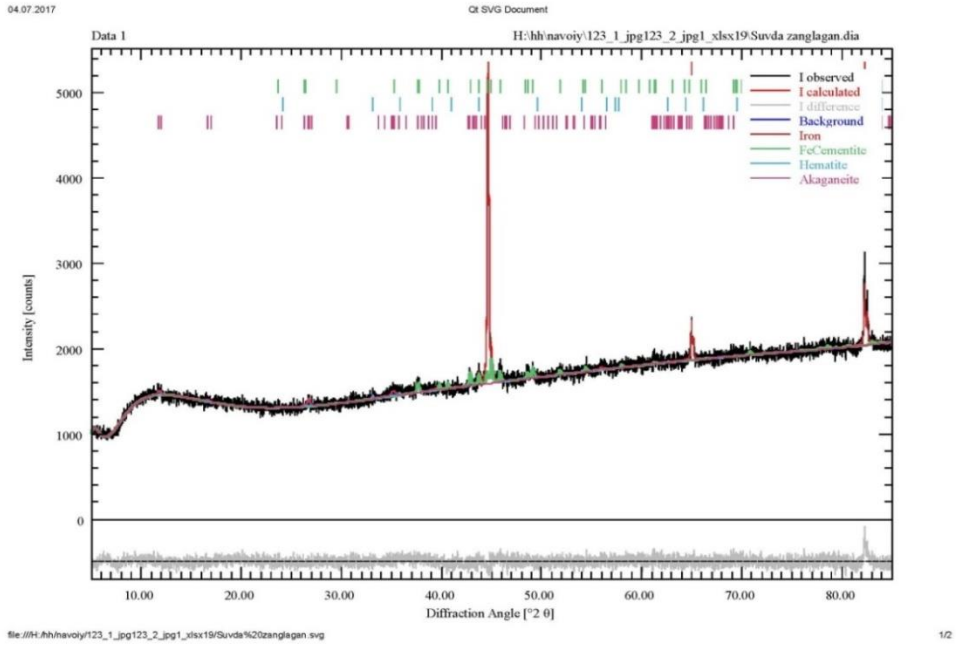

Fig.2. X-ray phase analysis by the Rietveld method on sample No. 2
stamps st. 3 in hydrochloric acid solution (pH = 2) for 24 hours at a temperature of 20 ^{0}C.

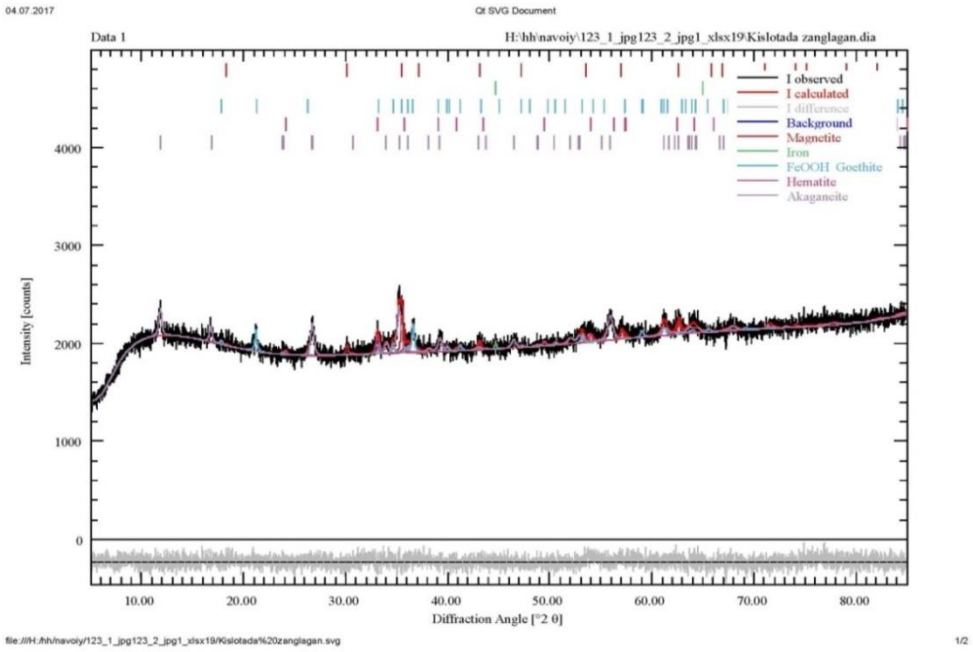

Fig. 3. X-ray phase analysis by the Rietveld method of sample No. 3. grade st. 3 in an alkaline solution (pH = 11) for 24 hours at a temperature of 20 ^{0}C.

Table 1.

The results of X-ray phase analysis of grade st. 3 in an aqueous solution (pH = 7) for 24 hours at a temperature of 20 ^{0}C.

Phase name	Quantity (%)	Accuracy (%)
Iron (Fe)	100	0

Table 2

The results of X-ray phase analysis of grade 3 in hydrochloric acid solution (pH = 2) for 24 hours at a temperature of 20 ^{0}C.

Phase name	Quantity (%)	Accuracy (%)
Iron (Fe)	54.1	1.3

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Cementite (Fe_3C)	31.0	1.2
Hematite (Fe_2O_3)	2.1	0.8
akagenite (β- FeOOH or $Fe_4O_8Cl_{0.675}$)	12.8	1.1

Table 3

The results of X-ray phase analysis of grade st.3 in an alkaline solution (pH = 11) for 24 hours at a temperature of 20 ^{0} C.

Phase name	Quantity (%)	Accuracy (%)
Iron (Fe)	0.5	0.1
Magnetite (Fe_3O_4)	11.7	0.6
Hematite (Fe_2O_3)	8.2	0.7
Goethite (α- FeOOH)	20.8	0.9
akagenite (β- FeOOH or $Fe_4O_8Cl_{0.675}$)	58.8	1.3

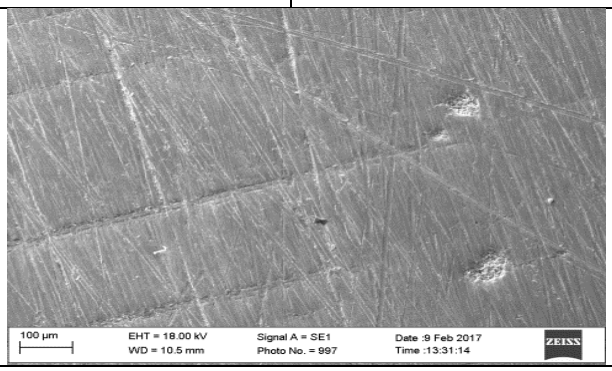

Fig.4. Pictures of sections of the surface of the sample, obtained on a scanning electron microscope steel grade st.3 in the presence of 0.02 wt. % IC-MK-21 in alkaline solution (pH = 11).

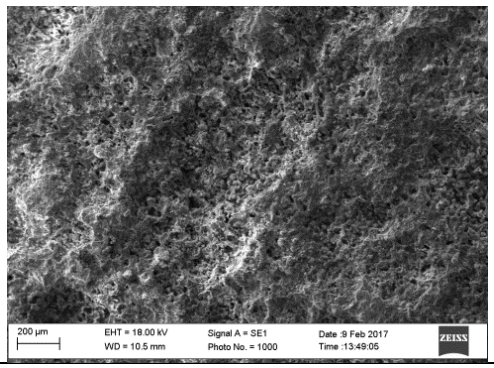

Fig. 5. Pictures of sample surface areas obtained on a scanning electron microscope, grade st. 3 in hydrochloric acid (pH =2).

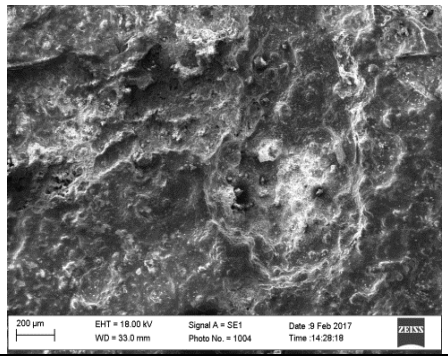

Fig.6. Pictures of sample surface areas obtained with a scanning electron microscope, Art. 3 in alkaline solution (pH =11).

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The results of X-ray phase analysis show that the surface of sample No. 1 consists of 100% iron, table.1 and an inhibitor prevents rust. An analysis of the surface of sample No. 1 on a scanning electron microscope also showed that the inhibitor protected the surface of the sample from the action of hydrochloric acid, but certain areas on the surface of the sample were exposed to it and corrosion results can be seen in these places (Fig. 5).

The surface of sample No. 2 consists of iron (Fe), cementite (Fe_3C), hematite (Fe_2O_3) and akagenite (β- FeOOH or $Fe_4O_8Cl_{0.675}$) (Table 2). Analysis of the surface of sample No. 2 on a scanning electron microscope shows that the surface of the sample has an uneven and porous structure (Fig. 4). Akagenite is an intermediate product of corrosion, and hematite (Fe_2O_3) is the final product. The presence of cementite in the sample can be explained as a product of the interaction of carbonates in water with iron.

The surface of sample No. 3 consists of iron (Fe), magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite (α-FeOOH) and akagenite (β- FeOOH or $Fe_4O_8CI_{10}$,) (Table 3). It is obvious that according to the X-ray penetration thickness, the main products of the corrosion process during the action of hydrochloric acid on the sample are goethite (α- FeOOH) and akagenite (β- FeOOH or $Fe_4O_8CI_{10}$) , ~ 80%, and also magnetite (Fe_3O_4) and hematite (Fe_2O_3) ~20%. On the last tab in Fig. 1, crystals of the formed akagenite are visible.

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