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CORROSION BEHAVIOR OF MILD STEEL IN AQUEOUS SOLUTIONS OF AMMONIUM NITRATE

The aqueous ammonium nitrate solutions exhibiting a strong general or local corrosion attack to mild and low alloy steels. The corrosion aggressiveness is higher than nitrate solutions of alkali and alkaline earth metals to the same steels, mainly due to lower pH and complexing properties of NH_4^+-ions, which facilitates the passage of Fe^{2+}-ions in the medium. Corrosion of iron and mild steel in NH_4NO_3 solutions have been studied by conventional methods and without providing information about the corrosion products and the probable reactions which control the corrosion processes. In the presented work, using electrochemical, physical, analytical and metallographic methods has been investigated the corrosion-electrochemical behavior of low-carbon mild steel (0.17% C) in aqueous solutions of ammonium nitrate. The corrosion-electrochemical parameters of the steel have been determined depending on the concentration, temperature and pH of the medium, as well the effects of thermal treatments – quenching and tempering at temperatures in the range of 200÷600°C. Under these conditions, the steel shows susceptibility or resistance to stress corrosion cracking (SCC). The corrosion products were determined by a Mössbauer analysis to correlate them with the corrosion behaviors of tested steels.

Keywords: Corrosion; stress corrosion cracking; mild steels; nitrate solutions; Mö-spectroscopy

1. Introduction

The aqueous ammonium nitrate solutions exhibiting a strong general or local corrosion attack to mild and low alloy steels [1-10]. Their corrosion aggressiveness is higher than that of alkali and alkaline earth metals nitrate solutions to the same steels, mainly due to lower pH and the complexing properties of NH_4^+-ions, which facilitates the passage of Fe^{2+}-ions in the medium [6].

Corrosion of iron and mild steel in NH_4NO_3 solutions have been studied by conventional methods and without providing information of the corrosion products, and the probable reactions which control the corrosion processes [4,5]. Some corrosion products because of the reaction between iron and NH_4NO_3 solution (44%) as related mechanisms resulting in magnetite formation have been studied [9, 10]. The authors have found that the magnetite on the sample surfaces has a passivating effect, and the Fe^{3+}/Fe^{2+} ratio increases with raising solution temperature. The effect of the time duration of the sample-medium contact, temperature and pH of the medium on the final corrosion products of electrolytic iron in 45% NH_4NO_3 aqueous solutions have been analyzed by means of Mö-spectroscopy, combined with XRD, IR and SEM techniques [11,12]. The Mö-spectroscopy is a reliable method providing both qualitative and quantitative information about the corrosion products of the iron and its alloys. An important advantage of this method is its equal sensitivity to both crystalline and amorphous compounds [12,13].

Despite many published studies, the literature sources do not provide a coherent point of view on corrosion kinetics and mechanisms with respect to either general or local forms of corrosion [14-20]. Moreover, the relationships between corrosion-electrochemical parameters and the final compositions of corrosion products are not well defined when resistances and susceptibilities of mild steels to SCC in nitrate media are at issue. This suggests a field of study and lines of investigations where there is still deficiency in information.

Using modern electrochemical and physicochemical analytical methods, the corrosion behavior of new, modern materials in various environments and conditions (composition, concentration, microstructure, microhardness and others) has been investigated [23-25].

In the presented work, using electrochemical, physical, analytical and metallographic methods has been investigated

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the corrosion-electrochemical behavior of low-carbon mild steel (0.17% C) in aqueous solutions of NH_4NO_3. The corrosion-electrochemical parameters of the steel have been determined depending on the concentration, temperature and pH of the medium, as well the effects of thermal treatments – quenching and tempering at temperatures in the range of 200÷600°C. The composition of the final corrosion products upon conditions where the steel is susceptible or not to SCC [14-22] is one of the principal tasks of this study.

2. Experiment

2.1. Material and samples

The chemical composition of low carbon hot forming steel is presented in TABLE 1 and is taken from the certificate of the purchased material.

TABLE 1

Chemical composition of the steel (% in mass)

C	Mn	Si	P	S	Cr	Ni	Cu
0.17	0.36	0.016	0.01	0.029	0.06	0.06	0.11

The mild steel samples were obtained as hot drawn rods. The steel has ferrite-pearlite structure, grade 7(8) and the carbon is concentrated on individual ferrite-pearlite grains or on the contact area of the ferrite grains – Fig. 1.

Thermal treatments of the steel samples covered water hardening from 930°C and tempering in an inert argon atmosphere in temperature range 200÷900°C. After these operations, the samples were left in the furnace in a pure argon atmosphere to be chilled down to room temperature (the procedures are described elsewhere [17]).

The tested samples/electrodes for polarization measurements were with a disk shape and operating surface area of 1.0 cm^2. The rest of the electrodes are insulated by means of polymeric material (duracryle).

Samples with rectangular form and working area 20.0 cm^2 were used for the determination of corrosion products by Mössbauer spectroscopy.

Preliminary treatments of all types of test specimens consider cleaning by sandpaper, washing with distilled water and degreasing with an alcohol-ether mixture.

2.2. Solutions

Working media are aqueous ammonium nitrate solutions with following parameters:

- concentration values of 1.0, 5.0 and 9.0 M,
- temperature values of 20.0÷90.0°C,
- pH values of 1.0, 2.9, 4.6 and 9.0.

The solutions are prepared from NH_4NO_3 (pa) and mono distilled water, acidified or alkalized respectively by HNO_3 (pa) or NaOH (pa).

2.3. Methods

Potentiodynamic polarization method. The experiments were carried out with EG&G Princeton Applied Research, Potentiostat/Galvanostat, Model 273 thus allowing to determine a variety of characteristics such as corrosion rate, corrosion potential etc. Moreover, the method allows determining many parameters characterizing the passive state such as a critical potential and a current, the potential and current of total passivation, the width of passivation zone.

Mössbauer analysis. By help of a standard Mössbauer spectrometer (the source is Co57 (Pd)) and the speed scale for all spectra is given in relation to the reference zero of α-Fe operating in a transmittance regime and a constant acceleration mode, the composition of the corrosion products was determined. The average sample thickness was about 10 mg cm^{-2} and the spectra were taken at room temperature (25±2°C).

Metallographic analysis. The measurements were performed by an optical metallographic microscope, model Epi-

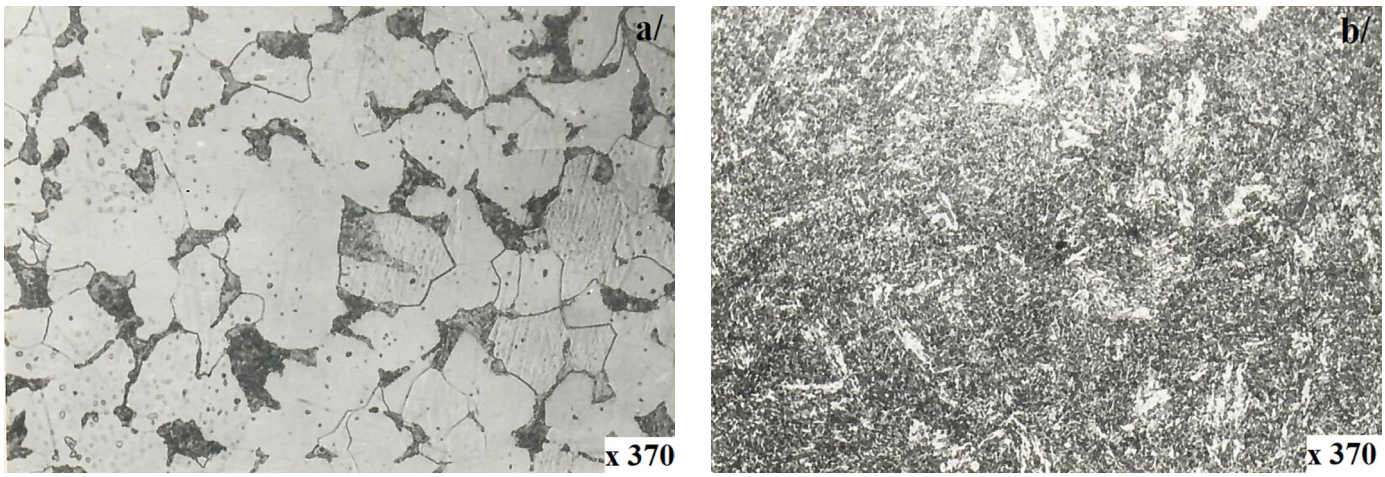

Fig. 1. Microstructure of the tested steel (×370): a/ as received; b/ quenching; c/ annealing at 200°C; d/ annealing at 600°C

typ-2, thus allowing seeing the effects of thermal treatments on steel microstructures as well as elucidating the natures of corrosion attacks when tests in different environments and conditions are carried out.

2.4. Results and discussion

2.4.1. Concentration and temperature effects

The main corrosion-electrochemical parameters of the mild steel tested in solutions with concentrations 1.0, 5.0 and 9.0 M (and temperature range 20÷90°C) were determined by potentiodynamic polarization method. The rate of potential change is 20 mV min^{-1} from a preliminary starting potential (–0.900 V vs. SCE) at which it remains unchanged for 3 min.

The potentiodynamic polarization relationships for mild steel tested in 9.0 M NH_4NO_3 aqueous solution are shown in Fig. 2. The plots reveal that the rise of temperature increases in the corrosion reaction rate. The change in the slope of the cathode curve at a temperature of 90°C is most likely connected with a reduced O_2 content and an increasing contribution of another depolarizer in the solution – NO_3^--ions.

The influence of temperature on the anodic part of the polarization curves is more noticeable. There is a delay in the transition of steel to a state of complete passivity with raising the temperature of the media. At the highest of temperatures studied (90°C), E_{cr} shifts again in the negative direction, –0.310 V (SCE). The critical current density i_{cr} increases by about 2 orders of magnitude as the temperature rises from 20°C to 75°C and decreases at 90°C.

The solution temperature affects strongly on the passivation potential, E_p and current in the passive state, i_p. More precisely, the values of E_p become more positive with increase in the temperature: from –0.390 V, SCE at 20°C up to about +0.300 V, SCE at 75°C and 90°C. The current density, i_p in the passive

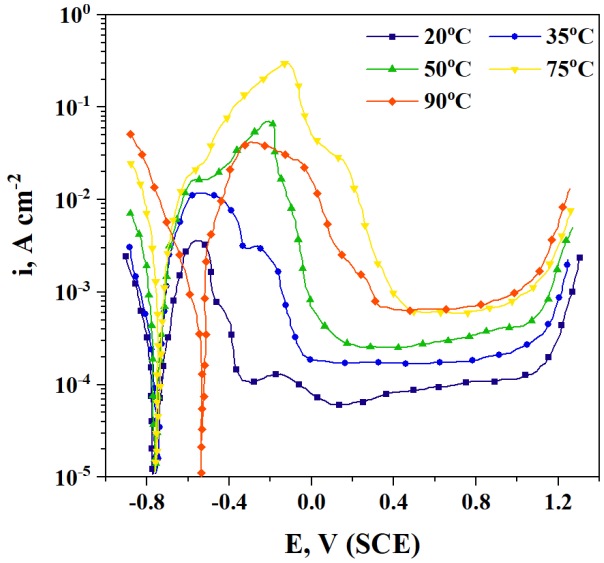

Fig. 2. Potentiodynamic polarization relationships E-lgi, obtained in 9.0 M NH_4NO_3

state increases as the temperature rises and at 75°C and 90°C (at these two temperatures the values of i_p are almost identical) is about one order of magnitude greater than in the case of 20°C. Similarly, as described above, the temperature effects on the critical values of potential and current were observed in the other two concentrations of ammonium nitrate solution – 1.0 M NH_4NO_3 and 5.0M NH_4NO_3.

The effects of both the temperature and solution concentration on the corrosion potentials and currents are shown in Fig. 3. The plots indicate that the value of E_{corr}, for all concentrations in the range studied, do not change significantly when the bath temperature rises to 75°C, but at 90°C it becomes more positive and increase at about twice. The latter is an indication that at this temperature there is more easy formation of a film by the corrosion products. The values of the corrosion current density, i_{corr} increase with the temperature up to 75°C, for all concentra-

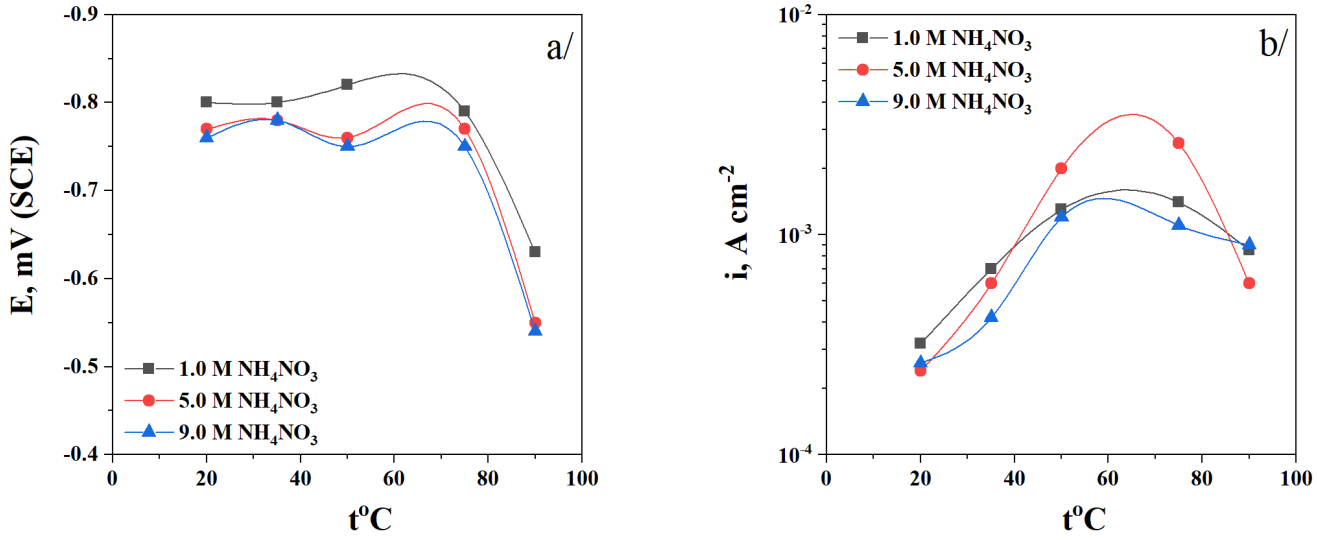

Fig. 3. Effect of temperature on corrosion potential, E_{corr} (a) and on corrosion current density, i_{corr} (b), obtained in NH_4NO_3 with different concentrations

tions of the test solutions studied, but they decrease at the highest medium temperature 90°C.

2.4.2. pH effect

The effect of pH on the corrosion-electrochemical parameters at 20°C and 90°C was studied in 5.0M NH_4NO_3, accepted as a model medium for stress corrosion cracking test of mild steels [16]. The relationships "*potential, E – current density, i*", shown in Fig. 4 are recorded upon a rate of potential change 20 mV min^{-1}. The main parameters are summarized in TABLE 2.

The data summarized in TABLE 2 reveal that the corrosion potential, E_{corr} in the system "mild steel – 5M NH_4NO_3" becomes more "noble" with rise in temperature for all pH values. More significant is the effect of pH on the corrosion current density i_{corr}. At pH = 1.0, i_{corr} has a higher value than those obtained at pH = 4.6 and especially at pH = 9.0, both at 20°C and at 90°C.

With increase in pH of the ammonium nitrate medium the steel transition to passive state is facilitated at both temperatures. The critical potential, E_{cr} becomes more negative with increase in pH, while the critical current density, i_{cr} decreases with about two orders of magnitude at pH = 9.0. The potential corresponding to complete passive state, E_p shifts towards negative values with increase in pH, while the density of the passivation current, i_p decreases upon the same conditions.

2.4.3. Effect of heat treatment

The effect of heat treatment of mild steel on polarization relationships „E-lgi" was conducted in 5.0 M NH_4NO_3, at temperatures 20°C and 90°C (Fig. 5). The steel specimens were thermally treated in the following consequent modes: quenching, quenching and tempering at 200°C, 400°C and 600°C. The corrosion-electrochemical parameters of thermally treated steel are summarized in TABLE 3.

The polarization curves and the related parameters reveal that the thermal treatment of the steel does not change largely its corrosion behavior. At both temperatures (20°C and 90°C) the corrosion potentials, E_{corr}, V (SCE) and the current densities, i_{corr}, [A cm^{-2}] are close in value. At a higher temperature of 90°C, however, the corrosion potentials are more positive, and the corrosion current densities are lower, compared to those obtained at a temperature 20°C. The highest temperature (90°C) delays the transition of the steel to a state of complete passivity and the values of both E_{cr}, V (SCE) and E_p, V (SCE) are more positive, while the ones of i_{cr}, A cm^{-2} and i_p, A cm^{-2} are greater than those determined at 20°C.

In general, the thermal treatment does not affect significantly on the corrosion-electrochemical parameters and from the polarization relationships E-lgi, cannot be judged about its strong influence on the susceptibility of the steel to stress corrosion cracking (SCC) in ammonium nitrate media [15]. Most likely, the effect of the internal stresses provoked by the thermal

TABLE 2

Corrosion parameters of mild steel in 5.0 M NH_4NO_3 at different values of pH

pH	*t*, °C	E_{corr}, V(SCE)	i_{corr}, Acm^{-2}	E_{cr}, V(SCE)	i_{cr}, Acm^{-2}	E_p, V(SCE)	i_p, Acm^{-2}
1.0	20	–0.710	1.10^{-4}	–0.180	8.710^{-2}	+0.180	9.110^{-5}
	90	–0.570	1.610^{-4}	–0.230	2.510^{0}	+0.340	9.010^{-4}
4.6	20	–0.680	2.410^{-4}	–0.540	8.210^{-3}	+0.110	5.210^{-5}
	90	–0.540	6.110^{-4}	–0.270	7.510^{-2}	+0.310	8.210^{-4}
9.0	20	–0.675	5.010^{-5}	–0.650	3.110^{-4}	–0.550	9.010^{-6}
	90	–0.500	7.110^{-5}	–0.470	6.410^{-4}	–0.380	3.410^{-5}

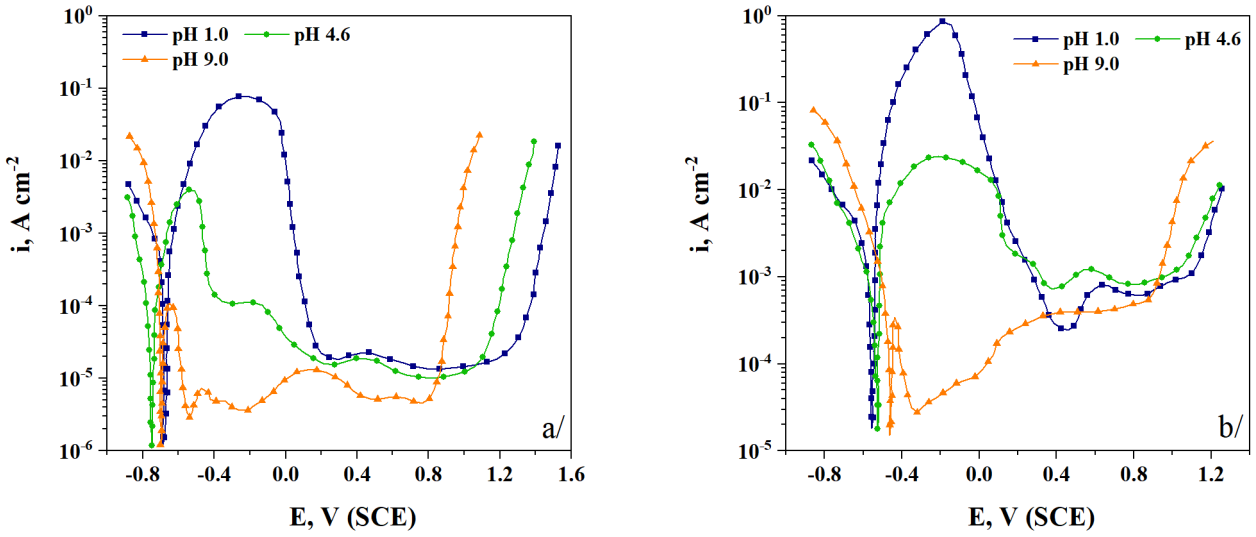

Fig. 4. Potentiodynamic polarization relationships E-lgi, obtained in 5.0 M NH_4NO_3: a/ 20°C; b/ 90°C

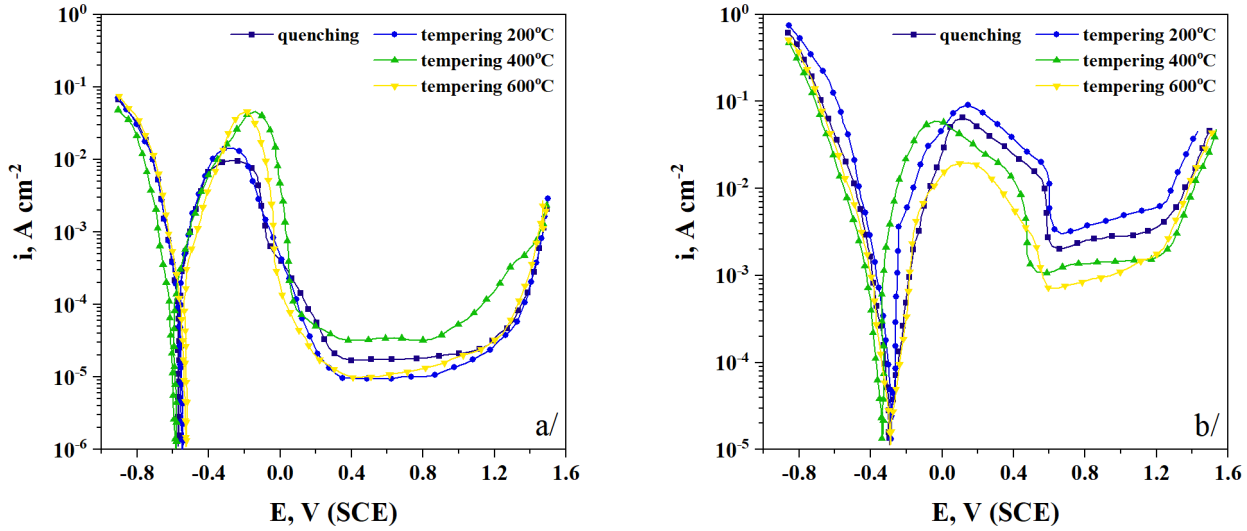

Fig. 5. Potentiodynamic polarization relationships E-lgi, obtained in 5.0 M NH_4NO_3: a/ 20°C; b/ 90°C

TABLE 3

Corrosion parameters of thermal treatment (TT) of mild steel in 5.0 M NH_4NO_3

TT	t, °C	E_{corr}, V(SCE)	i_{corr}, Acm^{-2}	E_{cr}, V(SCE)	i_{cr}, Acm^{-2}	E_p, V(SCE)	i_p, Acm^{-2}
quenching	20	–0.570	2.4×10^{-4}	–0.280	2.2×10^{-2}	+0.300	4.8×10^{-5}
	90	–0.280	4.8×10^{-5}	+0.070	1.3×10^{-1}	+0.620	3.2×10^{-3}
annealing, 200°C	20	–0.560	2.8×10^{-4}	–0.280	3.2×10^{-2}	+0.220	4.8×10^{-5}
	90	–0.280	5.0×10^{-5}	+0.130	1.0×10^{-1}	+0.620	3.2×10^{-3}
annealing, 400°C	20	–0.600	5.8×10^{-4}	–0.140	1.0×10^{-1}	+0.080	2.6×10^{-4}
	90	–0.310	5.2×10^{-5}	–0.040	1.1×10^{-1}	+0.520	2.0×10^{-3}
annealing, 600°C	20	–0.590	2.8×10^{-4}	–0.190	1.1×10^{-1}	+0.080	1.3×10^{-4}
	90	–0.290	4.0×10^{-5}	+0.140	4.0×10^{-2}	+0.570	1.5×10^{-3}
as received	20	–0.770	2.4×10^{-4}	–0.580	1.2×10^{-2}	–0.350	1.2×10^{-4}
	90	–0.540	6.0×10^{-4}	–0.300	9.0×10^{-2}	+0.320	2.0×10^{-3}

treatment may enhance the steel sensitivity to SCC. The lower resistance of the quenched steel with respect to SCC might be related to the higher level of stress in the low-carbon martensite. With increase in the tempering temperature up to 600°C, there is a reduction in internal stresses, the structure of the steel becomes more homogeneous, and thus its resistance with respect to SCC increases.

2.5. Nature of the corrosion products

By Mössbauer analysis, the composition of the corrosion products upon conditions allowing SCC as well as when a steel is resistant to this corrosion-mechanical failure in aqueous NH_4NO_3 solutions, were determined.

The Mössbauer spectra are taken down upon the following conditions: 1.0M and 5.0M aqueous solutions of ammonium nitrate with pH 1.0, 2.9, 4.6 and 9.0; at potentials –0.700, 0.00, +0.500 and +1.200 V, SCE. The bath temperature ranged from 20°C up to 90°C.

The Mössbauer spectra of corrosion products developed at the steel surface in 5.0 M NH_4NO_3 solution are shown in Fig. 6 at pH = 1.0 (a) and pH = 4.6 (b).

The influence of potential on the composition of the surface film of corrosion products, (through Mössbauer spectra), is shown in Fig. 7: –0.700 V, SNE (a) and +1.200 V, SNE (b).

The results from the corrosion product analysis after the Mössbauer spectra treatment are summarized in TABLE 4. Moreover, TABLE 4 contains also information about the corrosion products passed into the volume of the solution. The latter are related to greater extent to the general corrosion behavior rather than to SCC, and therefore henceforth only the composition of the film formed on the steel surface will be commented.

In 1.0 M NH_4NO_3 solutions at 20°C there is a stable corrosion film formed on the steel surface, while in 5.0M NH_4NO_3 solutions, at the same temperature, the film consists primarily of paramagnetic fractions of Fe^{3+} (~85%). At 90°C bath temperature, in both concentrations, there are formations of films consisting of Fe_3O_4 + γ-Fe_2O_3; in the 1.0 M solution its quantity is higher (>85%). All remaining components of the formed films are in paramagnetic state.

The effect of pH on the composition of corrosion products was determined only for tests performed in 90°C baths. At the metal surfaces, are formed films, which consist mainly of Fe_3O_4 + γ-Fe_2O_3. It is worth noting that with increase in pH the content

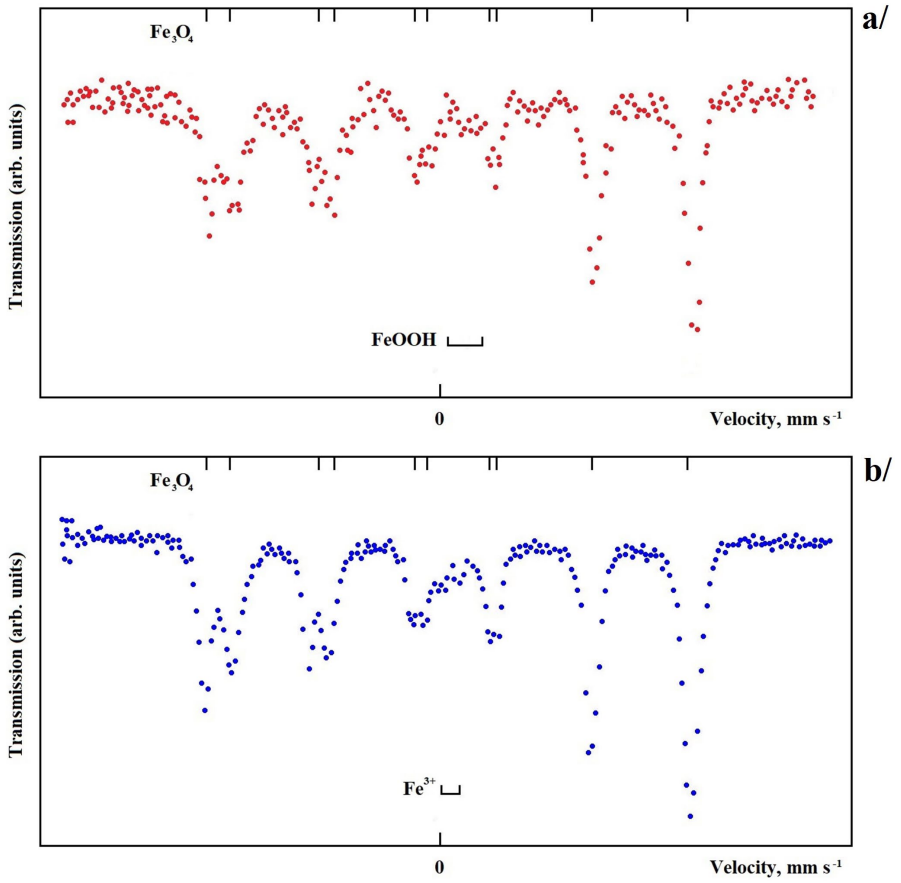

Fig. 6. Mössbauer spectra of corrosion products formed in 5M NH_4NO_3, 90°C: a/ pH = 1.0; b/ pH = 4.6

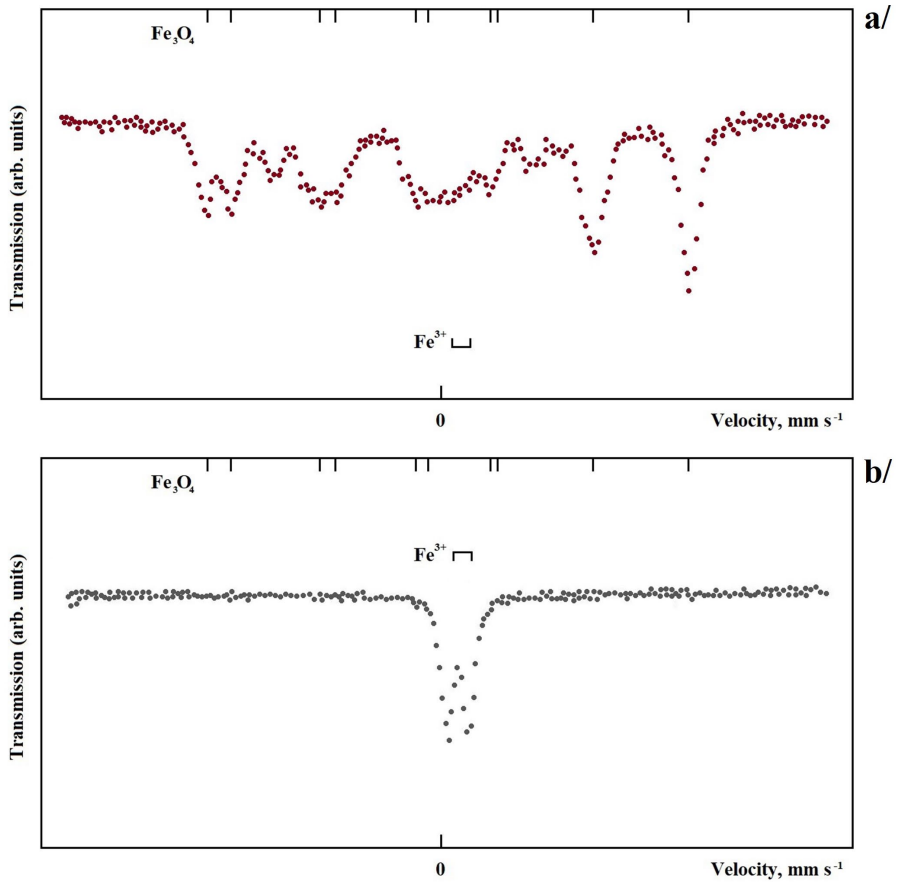

Fig. 7. Mössbauer spectra of corrosion products formed in 5 M NH_4NO_3, 90°C: a/ E = –0.700 V, SCE; b/ E = +1.200 V, SCE

TABLE 4

Mössbauer analysis of corrosion products of mild steel in ammonium nitrate aqueous solutions

Corrosion medium	*t*, °C	pH		E, V (SCE)		Place	Corrosion products
		initial	final	initial	final		
1.0 M NH_4NO_3	20	4.9	7.5	–0.590	–608	in solution	fm – Fe_3O_4~25÷27%, γ-Fe_2O_3~40%; pm – FeOOH, Fe^{3+}-22%
	90	4.9	7.04	–0.560	–405	on surface	fm – Fe_3O_4~85%, γ-Fe_2O_3; pm – Fe^{3+}~15%
						in solution	fm – Fe_3O_4 (less Fe^{2+})-mainly
5.0 M NH_4NO_3	20	4.6	7.2	–0.570	–589	on surface	pm – Fe^{3+}~75÷85%; FeO~20%
						in solution	fm – Fe_3O_4~18%, γ-Fe_2O_3~49%; pm – Fe^{3+}-22%, α-FeOOH~10%
	90	4.6	7.0	–0.555	–320	on surface	fm – Fe_3O_4~70%, γ-Fe_2O_3; pm – Fe^{3+}~2÷3%
						in solution	fm – (Fe_3O_4+γ-Fe_2O_3)~70%; pm – Fe^{3+}~15÷20%, α-FeOOH~12%
5.0 M NH_4NO_3	90	1.0	1.7	–0.508	–290	on surface	fm – (Fe_3O_4+γ-Fe_2O_3)~70%; pm – α-FeOOH
						in solution	fm – Fe_3O_4~15%; pm – Fe^{3+}-mainly
5.0 M NH_4NO_3	90	2.9	6.8	–0.545	–345	on surface	fm – (Fe_3O_4+γ-Fe_2O_3)~70%
						in solution	fm – Fe_3O_4-mainly; pm – Fe^{3+}~10÷11%
5.0 M NH_4NO_3	90	9.0	7.7	–0.194	–670	on surface	fm – Fe_3O_4+γ-Fe_2O_3
5.0 M NH_4NO_3	90	4.6	—	–0.700	–700	on surface	fm – (Fe_3O_4+γ-Fe_2O_3) – mainly; pm – Fe^{3+}~6÷7%
						in solution	fm – Fe_3O_4~25%, γ-Fe_2O_3+α-Fe_2O_3-basically; pm – Fe^{3+}~10%
5.0 M NH_4NO_3	90	4.6	—	0.0V	0.0V	on surface	fm – Fe_3O_4-traces; pm – FeOOH, Fe^{3+}-mainly
						in solution	fm – Fe_3O_4~20÷25%, α-Fe_2O_3; pm – Fe^{3+}~3÷4%
5.0 M NH_4NO_3	90	4.6	—	+0.500	+0.500	on surface	fm – Fe_3O_4-traces; pm – (γ-Fe_2O_3 or α-FeOOH)~32%, Fe^{3+}~68%
5.0 M NH_4NO_3	90	4.6	—	+1.200	+1.200	on surface	pm – Fe^{3+}-mainly
						in solution	pm – α-Fe_2O_3~10%, Fe^{3+}-mainly

Note: pm-paramagnetic phase; fm-ferromagnetic phase

of oxides with spinel structure increases up to 70% at pH = 1.0 and becomes 100% at pH = 9.0.

The determination of the corrosion products at the potential where the steel is resistible against the SCC (–0.700 V, SCE) as well as when there are tendencies to failure (0.00 V, 0.500 V, +1.200 V, SCE) reveal that: at –0.700 V, SCE the film is formed mainly of Fe_3O_4 and γ-Fe_2O_3. At the other potentials the spinel oxides are of negligible amounts (the dominating is the paramagnetic Fe_3^+ (α-FeOOH, γ-Fe_2O_3, etc.).

3. Conclusions

The tests of corrosion behaviour of widely applied mild steel (0.17% C) in aqueous ammonium nitrate solutions (NH_4NO_3) in the concentration range 1.0 M- 9.0 M, reveal that:

- The increase of the bath temperature from 20°C up to 90°C, in the range of concentration applied, shifts the corrosion potential toward more positive values, while the corrosion current density increases, reaching a maximum at about 75°C, followed by a decrease. The transition of the steel into a passive state delay and its stability decreases with rising of a medium temperature.
- With increase of pH, in case of 5.0 M NH_4NO_3 solution, the corrosion potential becomes nobler. This potential at low pH is about two-fold in order magnitude lower than when pH = 9.0. The increase in pH facilitates the steel transition into passive state.
- Quenching and tempering at 200°C, 400°C and 600°C of the sample tests do not reveal effects on the steel corrosion behavior as well as on the corrosion-electrochemical parameters.
- Upon all experimental conditions applied where the steel exhibits increased resistibility against SCC, i.e., in 1.0 M solutions, pH > 8.5, E = –0.600÷–0.800 V, SCE the formation of spinel oxides Fe_3O_4 and γ-Fe_2O_3 is dominating.

Acknowledgments

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