

Electrochemical Comparative Characteristics of La(Fe,Si)₁₃ Type Materials with Different Content of Co in Acidified Phosphate Environment in Presence of Cl⁻ Ions

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In the paper the electrochemical characteristics of alloys based on La(Fe,Si)₁₃ phase have been presented. The material for research was the LaFe_{11.8-x}Co_xSi_{1.2} alloy with different cobalt content. Presented electrochemical characteristics provide information about pitting corrosion. This kind of corrosion is one of the most common local corrosion, it is especially undesirable for multi-phase alloys based on rare earth elements, as it contributes to the sudden and unexpected destruction of the surface of the material in working conditions. In the paper the results of potentiokinetic corrosion tests determined for LaFe_{10.72}Co_{1.08}Si_{1.2} and LaFe_{11.00}Co_{0.8}Si_{1.2} alloys in acidified phosphate solution (pH = 3) with different addition of chloride ions (Cl⁻) have been presented. Also analysis of alloys surface before and after corrosion measurement have been presented. The surface analysis has been carried out on the basis of profilometric roughness tests and optical microscopy observation. The studies have shown low resistance of LaFe_{10.72}Co_{1.08}Si_{1.2} and LaFe_{11.00}Co_{0.8}Si_{1.2} alloys to pitting corrosion, which, however, increases with Co content increase.

Keywords: electrochemical characteristics, pitting corrosion, La(Fe,Si)₁₃, potentiokinetic curves, roughness measurement.

1. INTRODUCTION

The magnetocaloric effect (MCE) is defined as material ability to heat or cool depending on the magnetic field [1, 2]. Interesting property and the value of MCE has a compound Gd₅Si₂Ge₂ [3]. However, because of the high costs of obtaining material based on Gd₅Si₂Ge₂, it did not find wider application in everyday devices. An alternative for this material are alloys based on La(Fe,Si)₁₃ – the advantage is the relatively low cost of production, while the disadvantage is the low value of the Curie temperature (T_C). Therefore, the most important factor in alloy preparation is to generate sufficiently large volume fraction of the La(Fe,Si)₁₃ phase responsible for their unique properties. In Fe-based alloys, the LaFe₁₃ phase is metastable in the comparison to the LaCo₁₃ phase observed in Co-based alloys. Therefore, the studies on the use of alloys containing cobalt in its composition (La(Fe,Co,Si)₁₃) on a large scale are carried out. The research are aimed to change the chemical composition, which would effect on the value of the constant crystal lattice of studied alloys to resulting in T_C increase and therefore a wider application of material base on La(Fe,Co,Si)₁₃ [4–8].

An important property of the material durability under operating conditions is its resistance to corrosion process.

Pitting corrosion is one of the most common type of local corrosion. Pits occur in preferred places of the surface, especially located on inclusions, defects, dislocation estuary, grain boundaries, interphase boundaries. The pitting corrosion is difficult to identify due to the absence of visible corrosion products especially in the initial stage. The development and propagation of pitting often tend to have strong form and pose the threat of sudden failure of the material [9, 10]. Significant problem with materials based on the rare earth elements (RE = La, Nd etc.) is their corrosion susceptibility. It is a consequence of the presence of several dozen percent of rare earth elements in chemical composition. It is known that the RE elements belong to the most electrochemically active metals (e.g. $E_{La^{3+}/La} = -2.52$ V, $E_{Nd^{3+}/Nd} = -2.43$ V) [11]. As many authors indicate [12–14], in most acidified environments the material based on rare earth elements dissolves activationally, which is particularly undesired. However, in acidified phosphate solution, as well as in neutral and alkaline solutions the their effectively passivate [15, 16]. A passive layer consisting oxides and hydrated metal oxides forms on the alloy surface. Electrochemical durability of a passive layer depends on the pH of the solution and on the presence of ions in the environment. In the presence of halide ions, a passive layer is easily damaged and local corrosion (pitting corrosion) develops [15, 16].

The paper presents the electrochemical characteristics determined for modified materials based on La(Fe,Si)₁₃

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with different content of cobalt – comparison of $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ and $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$. The potentiokinetic tests have been carried out in 0,5 M phosphate solution acidified to $\text{pH} = 3$ with the presence of chloride ions (Cl^-). As part of the work was also to analyze the surface of the materials ($\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ and $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$) using an optical microscope and a contact profilometer.

2. EXPERIMENTAL

The material for the examination were La-Fe-Si alloys based on $\text{La}(\text{Fe},\text{Si})_{13}$ phase. Chemical composition is presented in Table 1. The evolution of microstructure and the phase constitution have been described in other papers [8, 17]. The study material was produced with the arc melting technique of pure components in a protective atmosphere Ar. In order to homogenize the composition, the samples were melted ten times. Then the samples were embedded in quartz ampoules under reduced pressure. Thus prepared materials were subjected to annealing in temperature of 1323 K by the time of 15 days, after which they were cooled rapidly in water [9].

Table 1. Chemical composition of examined type $\text{La}(\text{Fe},\text{Si})_{13}$ alloys – sample symbol

Alloy composition	Sample number
$\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$	(1)
$\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$	(2)

Electrochemical measurements were performed using electrochemical measuring station CHI 618a, at a potential scan rate of 0.01 V s^{-1} , using its offset from the cathode ($E = -0.8 \text{ V}$) to anode ($E = +0.8 \text{ V}$). The values of electrode potentials are expressed relative to the saturated calomel electrode (SCE) $E_{\text{SCE}}^0 = 0.2446 \text{ V}$ at 298 K. Potentiokinetic polarization curves were determined in 0.5 M phosphate solution of NaH_2PO_4 acidified to $\text{pH} = 3$ by addition of 0.5 M H_3PO_4 , and with the chlorides addition, $[\text{Cl}^-] = 0.01\text{M}$; 0.03 M and 0.1 M – chloride ion concentrations were obtained by the addition of 1.0 M NaCl (Table 2). Extrapolation of the straight sections above potential breakthrough to the intersection of the line of minimum current allowed to determine the potential of perforation (E_{pit}). The tests have been performed at $20 \text{ }^\circ\text{C} \pm 0.1 \text{ }^\circ\text{C}$ at a rotational speed of the working electrode 21 rps. in de-aerated solutions. A pitting nucleation potentials have been determined on the basis of the one-way waveforms polarization.

Table 2. Types of the used corrosive media

Sample	0.5 M phosphate solution ($\text{pH} = 3$)			
	without additive	+ 0.01M Cl^-	+ 0.03 M Cl^-	+ 0.1M Cl^-
(1)	x	x	x	x
(2)	x	–	x	–

3. RESULTS AND DISCUSSION

It is known from the literature [12–14, 18–20] that alloys containing rare earth elements are very susceptible to corrosion. This is due to the high affinity for oxygen – in

the presence of a small amount of oxygen and moisture the alloys containing, for example, La, Nd, Sm, easily oxidizing [11, 14, 21], but the thermodynamic stability of the product on the surface does not constitute a barrier to further degradation of the material. Corrosion stability is perceived as a critical parameter. The corrosion behavior of this materials drastically affects the performances of magnetocaloric effect and lifetime. It is important to note that the production of anti-corrosion coatings on a La-Fe-Si type materials surface is not a satisfactory solution, since such coatings negatively affect thermal barriers, which can lead to cracks due to volume changes [19, 22]. The method that beneficially affect on corrosion resistance is to change the chemical composition in such a way that the passive layers formed on the surface will be more stable and the same has better protective properties. The changes in chemical composition based on the effect of galvanic coupling between the various phases. The small amounts of Mn, Co and C, have an effect on the corrosion rate, and also arranges operating temperature of alloys based on $\text{La}(\text{Fe},\text{Si},\text{X})_{13}$ [17, 23, 24].

In Fig. 1 the polarization curves for alloys with the composition $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ (sample 1 and 2) determined in 0.5 M phosphate solution with $\text{pH} = 3$ have been presented. Corrosion potential of the two samples is similar and is $E_{\text{corr}} = -0.80 \text{ V}$ resp. SCE. In terms of anodic potentials $> 0.4 \text{ V}$ a sharp decline in the value of the anodic current density ($i_{\text{pass}} \approx 5 \cdot 10^{-4} \text{ A/cm}^2$) has been observed. Similar tendency to active dissolution without a transition to passivation up to 0 V has been shown by Hu J. et al [24] for measurements in distilled water. Most of the tests conducted on such alloys are carried out in buffer solutions in the pH range of 4–8 [25], but according to Zhang et al. [26] and Wolff et al. [27] researches even after long term immersion in alkaline solution the pitting corrosion is not observed or are observed in small amounts [24]. This paper presents the results of measurements carried out in a strongly acidified phosphate solution ($\text{pH} = 3$) – the selection of the test solution has been driven by the fact that the alloys containing rare earth elements in phosphate solutions with this pH value easily passivate and the passive layer is made of hard-soluble phosphate compounds and even though the local corrosion (pitting corrosion) causing material damage [11, 14, 15, 25]. In the worldwide literature studies on the effect of chloride ion concentration on the stability of the passive layer on La-Fe-Co-Si alloys have not been found.

In Fig. 2, the polarization curves for sample 1 in 0.5 M phosphate solution ($\text{pH} = 3$) with different contents of chlorides (Cl^-) are presented. In the presence of a small concentration of Cl^- ions the breakdown of the passive layer has been observed. At a concentration of Cl^- ions, about 0.01M the breakdown potential is $E_{\text{pit}} = 0.39 \text{ V}$. Increasing the concentration of Cl^- ions in phosphate solution, contributed to the reduction of the E_{pit} (Table 3) and an increase in the anodic current density.

Table 3. The breakdown potentials for $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ alloy (sample 1) in acidic phosphate solution ($\text{pH} = 3$)

E_{pit}	0.5 M phosphate solution ($\text{pH} = 3$)		
	with 0.01M Cl^-	with 0.03 M Cl^-	with 0.1 M Cl^-
	+ 0.39 V	+ 0.15 V	–0.012 V

For comparison of resistance to pitting corrosion of the materials ($\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ and $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$), the potentiokinetic polarization curves in phosphate solution ($\text{pH} = 3$) with $[\text{Cl}^-] = 0.03 \text{ M}$ were performed (Fig. 3).

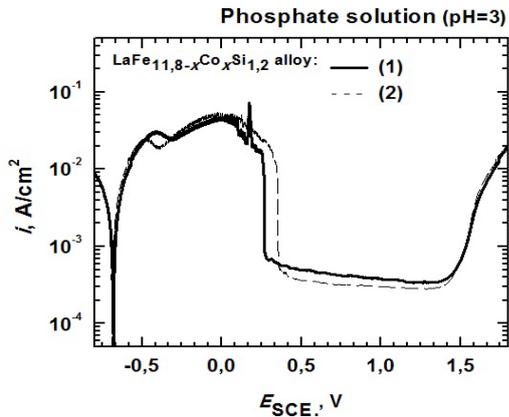


Fig. 1. Potentiokinetic polarization curves for $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ alloy in 0.5 M phosphate solution: (1) – $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$, (2) – $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$ ($\text{pH} = 3$, $20 \text{ }^\circ\text{C}$, $0.01 \text{ V}\cdot\text{s}^{-1}$)

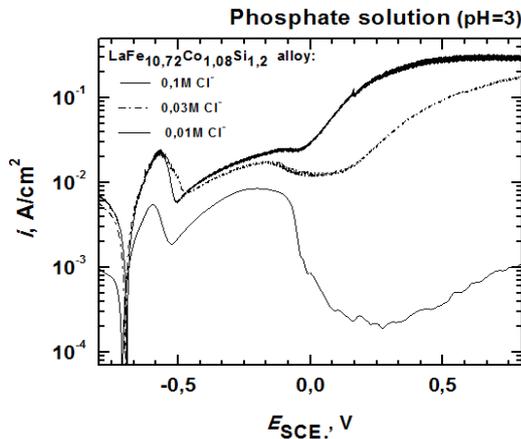


Fig. 2. Potentiokinetic polarization curves for $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ alloy (1 – $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$) in 0.5 M phosphate solution with chlorides ($\text{pH} = 3$, $20 \text{ }^\circ\text{C}$, $0.01 \text{ V}\cdot\text{s}^{-1}$)

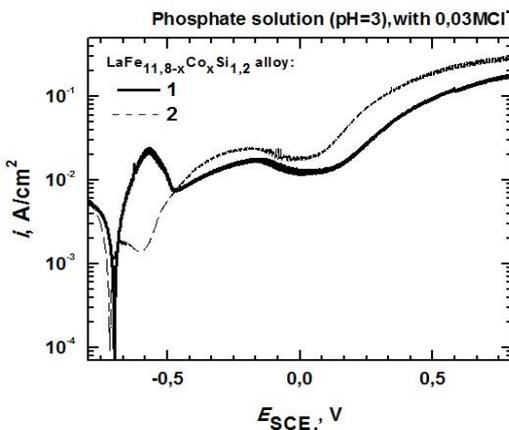


Fig. 3. Potentiokinetic polarization curves for $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ alloy ((1) – $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$, (2) – $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$) in 0.5 M phosphate solution with 0.03 M Cl^- ($\text{pH} = 3$, $20 \text{ }^\circ\text{C}$, $0.01 \text{ V}\cdot\text{s}^{-1}$)

In Fig. 1 it can be observed that the passive range for sample 2 ($\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$) is slightly smaller and range is

$0.45 \div 1.40 \text{ V}$, whereas for sample 1 ($\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$) is $0.30 \div 1.30 \text{ V}$. The attempt to achieve corrosion protection of the basic alloy (La-Fe-Si) by generating thin phosphate conversion coatings has not been so effective and the stability of the passive layer is adjusted by the pH of the solution [25]. It has also been observed that a change in the concentration of chloride ions significantly affects the corrosion characteristics of the material (Fig. 2 and Fig. 3). It can be seen that increasing the cobalt content in the chemical composition of the alloy increases the thermodynamic stability of the passive layer, as evidenced by the values of pitting potential (E_{pit}) – Table 4. In Fig. 3 it can be observed that the course of anodic curve for $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$ indicates a much easier transition into a passive state – after exceeding the corrosion potential, and critical passivation current ($i_{c, \text{pass}}$) is not observed (what can be noted on the material for the $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ material). However, $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$ in comparison to $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ is characterized with significant (almost double) reduction of the E_{pit} value (Fig. 3, Table 4) – smaller number of pits on a material containing a larger amount of Co also has been observed in Hu J. et al research [24].

Table 4. Values of breakdown potentials for $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ alloy ((1) – $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$, (2) – $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$) in acidic phosphate solution ($\text{pH} = 3$)

0.5 M phosphate solution ($\text{pH} = 3$) with 0.03 M Cl^-	
Sample	$E_{\text{pit}}, \text{ V}$
(1)	+ 0.15
(2)	+ 0.08

The comparative study of the sample surface before and after corrosion test has been carried out using an optical microscope with EPI 3D analyzer. On the $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ material surface (1) after exposure to 0.5 M phosphate solution containing 0.03 M Cl^- ($\text{pH} = 3$) a plurality of pits (Fig. 4 b) of different sizes have been observed.

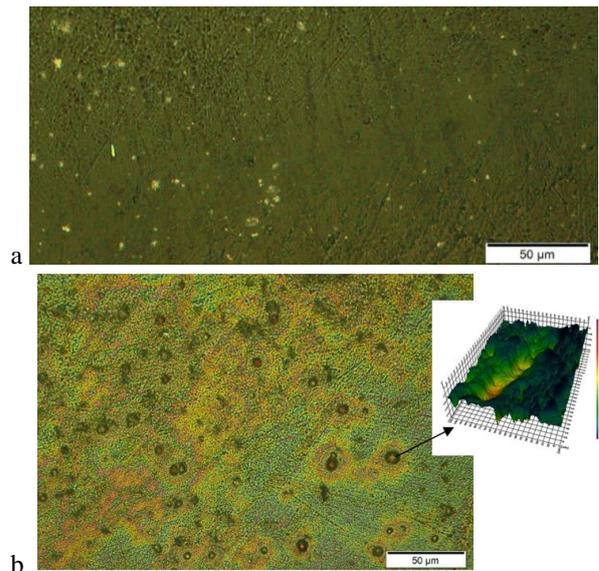


Fig. 4. Images of the 2D surface made an optical microscope for sample 1 ($\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$): a – before exposure; b – after electrochemical measurement of the material in a phosphate solution containing 0.03 M Cl^-

However, the contact of the $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ material with an aggressive environment has also led to a significant development of the surface (Fig. 5 b).

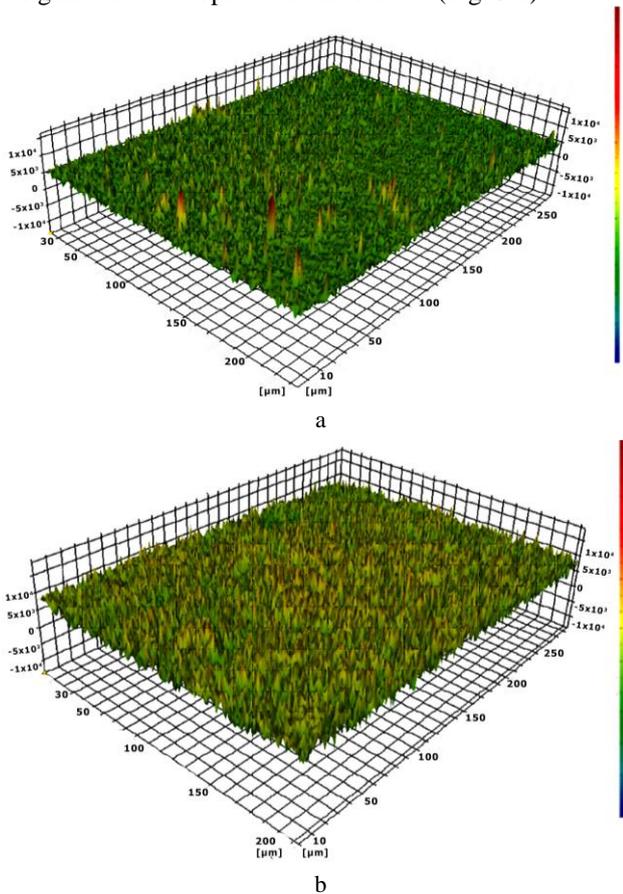


Fig. 5. The 3D surface images (magnification 50x) prepared with optical microscope with EPI 3D analyzer ($\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$): a – before exposure; b – after electrochemical measurement of the material in a phosphate solution containing 0.03 M Cl^-

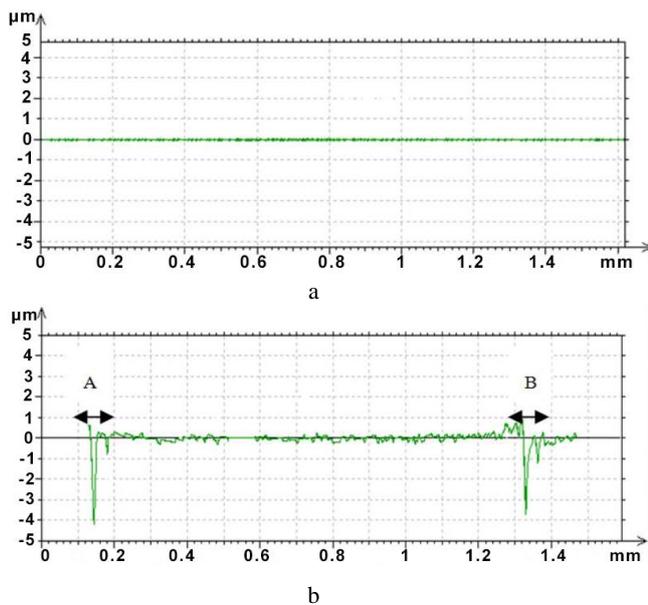


Fig. 6. Profilogram for $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ alloy before and after electrochemical measurement in 0.5 M phosphate solution containing 0.03 M Cl^- (sample 1): a – profilogram of surface before exposure; b – profilogram of surface after exposure

To analyze depth and the height of the material surface before and after exposure to 0.5 M phosphate solution with the addition of 0.03 M Cl^- , the 2D/3D images in the characteristic place have been taken. In representative section of the profile, the measurement of material loss into the cavity has been determined (Fig. 6 b). The results for the two sections A and B (Table 5) indicate a significant local area loss in the material after exposure to a corrosive medium.

As part of the research, also the surface roughness profiles have been obtained. The roughness profiles have been determined for the samples before and after exposure in a phosphate solution ($\text{pH} = 3$) containing 0.03 M of Cl^- . The results of the roughness measurements were processed by compatible TalyProfile Lite software. The analysis showed changes in the geometric parameters describing the surface structure. Changes occur in distribution the ordinate of the structure. According to this distribution (Fig. 6), after the corrosion process in the material (1) there is larger share of vertices/valleys in the deeper layers of the material.

Table 5. Selected height of profile for the $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ material surface (sample No. 1) before and after electrochemical measurement in phosphate solution containing 0.03 M of Cl^- ions ($\text{pH} = 3$)

Surface roughness parameters		A	B
	length	0.095 mm	0.0463 mm
before exposure	height difference	0.730 μm	0.11 μm
after exposure	height difference	5.73 μm	4.95 μm

4. CONCLUSIONS

On the basis of the presented results, it can be concluded that $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ type alloys with magnetocaloric properties passivate in phosphate solutions acidified to $\text{pH} = 3$, also in the presence of small concentrations of chloride ions. However, the passive layer is not an effective protective barrier for the surface of these alloys, in the presence of chloride ions the passive layer loses its electrochemical stability and after exceeding the pitting potential drastically develops the pitting corrosion. The study shows that the $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$ alloy indicates a much easier transition into a passive state - after exceeding the corrosion potential, and critical passivation current ($i_{c,pass}$) is not observed. The resistance of $\text{LaFe}_{11.8-x}\text{Co}_x\text{Si}_{1.2}$ material to pitting probably depends on the content of cobalt. Tested alloy $\text{LaFe}_{11.00}\text{Co}_{0.8}\text{Si}_{1.2}$ in comparison to $\text{LaFe}_{10.72}\text{Co}_{1.08}\text{Si}_{1.2}$ is characterized with significant (almost double) reduction of the E_{pit} value.

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