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Electrochemical influence of the nature and composition of halides on Al-12Si

S. Amokrane · M. A. Ladjouzi

Abstract The aluminium alloy Al-12Si has been polarized by potentiodynamic method at 25 °C under magnetic stirring and in an aerated solution. Its electrochemical behaviour was tested first by varying the concentration of NaI or NaCl (10^{-4}, 10^{-3}, 10^{-2}) added respectively to NaCl or NaI (10^{-3} M), and the pH of NaCl 10^{-3} M (pH=2.3, 7.3, 10) when adding HCl or NaOH (i.e. the composition of the solution), then by incorporating different ions familiar to an industrial atmosphere (Cu^{2+}, Zn^{2+}, SO_4^{2-}, NO_3^-, PO_4^{3-}) at 10^{-6} M to NaCl 10^{-3} M (i.e. the electrolyte nature). The use of the electrokinetic curves obtained allowed the access to the passivation (i_{pass}, E_{rup} and E_{rep}) and to the electrokinetic parameters (i_{corr}, R_p and P). They prove the behaviour dependence of the above alloy on the composition and nature of the electrolyte.

Keywords Aluminium alloy · Corrosion · Halides · Polarization

Introduction

Previously, aluminium was a metal not much appreciated because of some inferior properties compared to other ones.

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Nowadays, by exploring it deeper, these properties have been improved considerably, mechanical and chemical properties, particularly as an alloy thus increasing its used fields.

It should not be ignored how much the electrochemical behaviour of a material could be very different from one solution to another. For example, the same metal can be passivated in one solution, but not in another one. This can happen spontaneously in contact with air (such as Al) or by imposed current only. Its resistance with respect to the corrosion can be estimated by calculating the electrokinetic parameters such as i_{corr} and R_p.

In order to study the electrokinetic behaviour of aluminium alloyed with silicon Al-12Si (very frequently used in the building area for example), the latter has been tested by varying the composition (in terms of concentration and pH) and the nature (in terms of different ions) of the electrolyte, and polarizing such a system formed, in a potentiodynamic way.

The analysis of the curves obtained allowed us to determine and compare the corresponding parameters that were discussed before; the main conclusions could be reached.

Materials and methods

To realise our experiences, we use a cell into which we introduce a classical saturated calomel reference, a graphite auxiliary (of big area) electrode and the test electrode Al-12Si. The latter an Algerian product whose fluorescence X has revealed the composition: 87.6% Al+12% Si +0.14% Fe+0.26% of other element traces. The working electrode has been prepared in laboratory according to the next Fig. 1.

The test electrode is pre-treated (chemical degreasing, mechanical polishing and multiple rinsing) as many times as necessary to obtain a uniform and smooth area of the
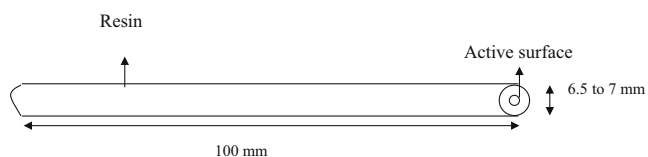
Resin

Active surface

6.5 to 7 mm

100 mm

Fig. 1 The working electrode

product which is controlled by metallographic analysis in order to ensure an optimal reproducibility of the results.

These three electrodes are immersed in an electrolyte constituted of regents of various origins (Fluka, Merck, Riedel-de-Haën and Prolabo) with purity ≥98%, diluted in bi-distilled water.

To study the variation of the pH, to the NaCl 10^{-3} M aqueous solution was added either HCl or NaOH to obtain a pH=2.3, 7.3 and 10. To study the effect of the industrial type ions we add CuCl$_2$, ZnCl$_2$, Na$_2$SO$_4$, NaNO$_3$ and Na$_3$PO$_4$ at 10^{-6} M to NaCl 10^{-3} M to keep conditions close to natural or spontaneous corrosion.

The pre-described cell is related to a measuring chain of EG&G type including a potentiostat/galvanostat (model 173), an electrometer (model 178), a computer and a current transformer. This chain is coupled to a current recorder (Sefram-TGM 164) and to a millivoltmeter with high impedance. The whole measuring elements constitute a circuit for potentiokinetic measures more useful for this type of investigation.

We start measuring in open circuit the free stationary potential (E_{stat} or E_{corr}) characterised by a stable potential of final portion in the E=f(t) curve. This E_{stat} value allows one to determine the extend of the studying potential, it has been estimated here at ±2 V on both sides from E_{stat}. The rate scanning of the potential was appreciated to be 1 mV/s.

In the case where a passivation steady-state occurs in the anodic portion of the curve, the reverse curve is registered to deduce the real repassivation potential (E_{rep}). The polarization is ensured in the anodic (E>E_{stat}) then cathodic (E<E_{stat}) way, firstly near equilibrium (the over voltage $\eta = E_i - E_{stat} \leq 10$ mV) by fixing the potential E_i with successive steps of 2 mV. We register the corresponding current density once the stability is reached: it results the i=f(E) curve whose the reverse slope is equal to the polarization resistance R_p. Then far from equilibrium ($\eta \geq 100$ mV), which allows us to draw the log(i)=f(E) curve, necessary from one hand to establish the anodic (b_a) and cathodic (b_c) Tafel slopes to deduce graphically or by calculation (Stern and Geary relation) the corrosion current density (i_{corr}) itself necessary to calculate the penetration depth of the pits (P), then from the other hand to determine the parameters linked with the passivation : passivation current density (i_{pass}), breaking potential (E_{rup}) and steady-state length (L_{pass}) in addition to E_{rep} mentioned above.

Results and discussion

The influence of the Cl$^-$ and I$^-$ concentration on the Al-12Si/NaI (10^{-3} M) and Al-12Si/NaCl (10^{-3} M) systems is given in Figs. 2 and 3, respectively.

The cathodic portions (left hand side curves) present profiles with no deviation (one slope), which is characteristic of a unique reaction, the monoelectronic reduction of water [1] since the pH is initially close to neutrality increases towards values ≥7.

In general, the cathodic current density increases with the concentration of Cl$^-$ or I$^-$ ions. Thus, the cathodic reaction produces more hydroxyl ions. That confirms the more and more alkaline pH as Cl$^-$ and I$^-$ increase.

To locate the Tafel area that predict, in theory, linear slopes (b_a and b_c) in the log(i) = f(E) curve, but which, in practice, deviate more or less from linearity, because of the kinetic mechanisms, and the simultaneous competitive phenomena with regard to the reaction process considered, such as the specific adsorption of species produced by solvent or the chemicals, the superficial films, sometimes of less conduction and therefore with a delayed effect on the kinetic overall rate [2], many options arise :

– Till ±20 mV on both sides from E_{corr} [3] the potential is not large enough to neglect one of the two current densities (i_{ox} or i_{red}) with respect to i_{total}.
– Above 100 mV [4] if the approximation $i_T \approx i_{ox}$ or i_{red} (according to the way of the polarization) is valid, the comparison between graphical i_{corr} (i_{corr} (*graphic*)) and calculated i_{corr} (i_{corr} (*S-G*)) does not give a satisfactory correlation.
– A compromise, already adopted by other authors [5–7] consists in locating the Tafel area at about ±50 mV on both sides from E_{corr}. Under these conditions, the results were found more satisfactory.
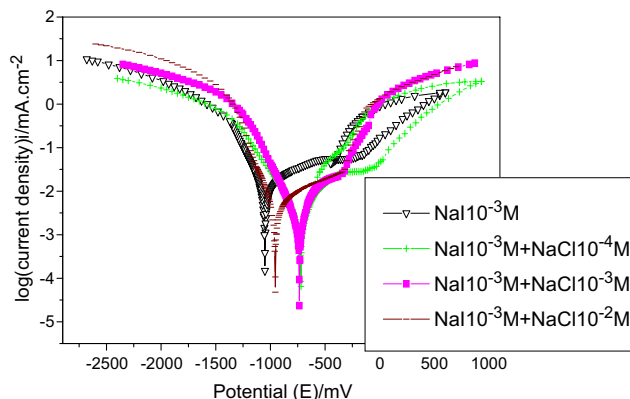

Fig. 2 Total polarization curves of the alloy Al-12Si in the considered medium: influence of Cl$^-$
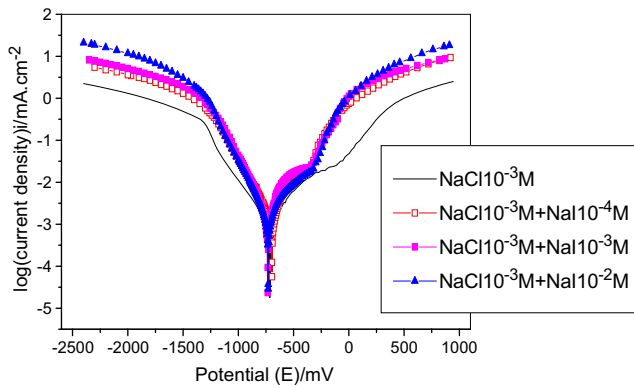
**Fig. 3** Total polarization curves of the alloy Al-12Si in the considered medium: influence of I^-

Most of the deduced values of b_c were close between them and to the theatrical one (−120 mV/decade) which means a pure activation kinetic.

The anodic process seems therefore more sensitive to the variation of the concentration. Using NaCl or NaI then adding I^- or Cl^- respectively with variable quantities does not influence significantly either the passivation parameters, nor the values of b_a, except at the more concentrated solution (10^{-2} M) for both electrolytes NaI 10^{-3} M+NaCl 10^{-2} M and NaCl 10^{-3} M+NaI 10^{-2} M where b_a becomes larger. At this concentration, the adsorption due to Cl^- and I^- becomes greater thus exerting a great delay on the kinetic process. This gap in b_a relative to the ideal theoretical value (30 mV/dec or 60 mV/dec if z_a=2 or 1 respectively) can be explained in terms of distortions in potential caused by the adsorption of chemical species and/or the formation of the spontaneous hydroxide film thickened by layers (or coatings) even unstable. The work of Pyun and Lee [8] proved that a repassivation occurs at the considered concentration of Cl^-. Besides this result is confirmed by the couple.

Al-Kharafi/Badawy [9] who establish that the degradation rate of the oxide film is very weak compared to that of the repassivation. Our results confirm the repassivation effect as described by these authors when there is only one halide, but not both of them (NaCl+NaI). In the mechanism proposed by Pyun and Lee, the presence of Si in the alloy seems to favour the formation of the oxide passivating film:

$$Al(OH)_2Cl + H_2O \rightarrow Al(OH)_3 + H^+ + Cl^-$$
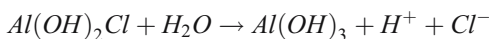
Rather than the direct formation of the chloride salt according to the reaction:

$$[AlCl_{ads}] + 2Cl^-_{aq} \rightarrow AlCl_3 + 2e^-$$
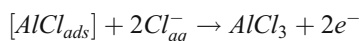
All R_p values displayed in our tables are obtained from graphs similar to Fig. 4. They all check the linearity of i=f(E) according to a law of Ohm type.

The simultaneous use of the b_a and b_c slopes and of R_p gives access to a second value of i_{corr}, calculated from the

Stern and Geary equation and which can be compared to that graphical when exploiting two distinct fields of study. Finally, the i_{corr} value can also be used in the empirical relation

$$P = 3267.98 . i_{corr} M / Z\rho$$

Where: 3267.98=empirical coefficient of corrosion (in s/C); i_{corr}=current density of corrosion; M=atomic weight (g); z=electrovalence; ρ=voluminal mass (g/cm^3) and P=penetration depth of the pit (cm)] to calculate P. All of these electrokinetic and passivation parameters are presented in Table 1.

Though in good agreement with each other, graphical and calculated i_{corr} present sometimes some discrepancy due to the imprecision in some values of b_a whose slopes are masked in the Tafel area by the early passivation flat appearing there.

We should note that the parameter P is in a perfect harmony with R_P, which is obvious since P is related to i_{corr} by constant terms for a given system, and i_{corr} itself is related to R_P. The less the latter is, the deeper is the pit.

The second variable, after the concentration, in the composition of the electrolyte is the pH in this study. The log (i)=f (E) curves corresponding to the three values of the pH studied (2.3, 7.3 and 10) are illustrated in Fig. 5; and the parameters deduced from their exploitation are given in Table 2.

The cathodic process depends on pH. Indeed, in the acid curve, the cathodic current density is higher than the others for the same over voltage. The profiles are increasing continuously with a monoelectronic reduction of water for the neutral and alkaline pH, and a protonic and monoelectronic discharge for the acid medium. The b_c slope, mainly for the acid pH is far from the theoretical one (−120 mV/dec) which is synonymous with a pure transfer, because of the presence of the hydroxide formed spontaneously, and which, in
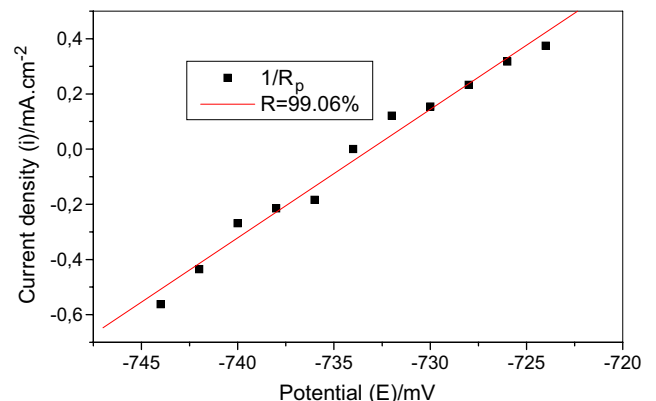

Fig. 4 Example of the variation of (current density versus the potential) i=f(E) (for the system Al-12Si/NaCl 10^{-3} M+NaI 10^{-3} M). Access to R_p

Table 1 Electrokinetic and passivation parameters deduced from the polarisation of the Al-12Si alloy in NaI (10^{-3} M) +NaCl (xM) and NaCl (10^{-3} M) +NaI (xM)

Electrokinetic Parameters	NaI 10^{-3} M	NaI 10^{-3} M+ NaCl 10^{-4} M	NaI 10^{-3} M+ NaCl 10^{-3} M	NaI 10^{-3} M+ NaCl 10^{-2} M	NaCl 10^{-3} M	NaCl 10^{-3} M+ NaI 10^{-4} M	NaCl 10^{-3} M+ NaI 10^{-2} M
E_{corr}(mV/ECS)	−1050	−720	−735	−955	−715	−695	−730
$-b_c$(mV/dec)	162.60	168.35	154.32	144.93	185.53	174.82	162.6
b_a(mV/dec)	324.87	177.62	175.13	294.98	183.15	189.03	222.72
i_{corr}(graphic) (μA.cm^{-2})	7.43	1.4	1.5	2.71	0.62	1.32	1.2
i_{corr}(S-G) (μA.cm^{-2})	9.76	1.99	1.66	3.21	0.76	2.13	1.8
R_p(Ωcm^{-2})	4.85	18.87	21.5	13.12	52.35	18.52	22.73
P(10^3 cm)	82.53	15.62	16.69	30.13	6.87	14.68	13.38
i_{pass}(μA.cm^{-2})	54.83	27.67	16.59	11.51	15.06	7.74	6.44
L_{pass}(mV)	240	207	230	490	300	212	280
E_{rup}(mV/ECS)	−280	−143.17	−350	−325.45	−108.22	−338.44	−314.73
E_{rep}(mV/ECS)	-400	–	–	–	–	–	–
pH before polarisation	6.15	6.25	6.2	6	6.05	6.35	6
pH after anodic polarisation	7.2	6.65	6.9	6.65	6.8	6.75	6.9
pH after cathodic polarisation	7.15	7.55	7.7	8.75	6.95	8.05	8.4

place of producing H$_2$ according to the protonic reduction, reacts with H$^+$ to form Al^{3+}:

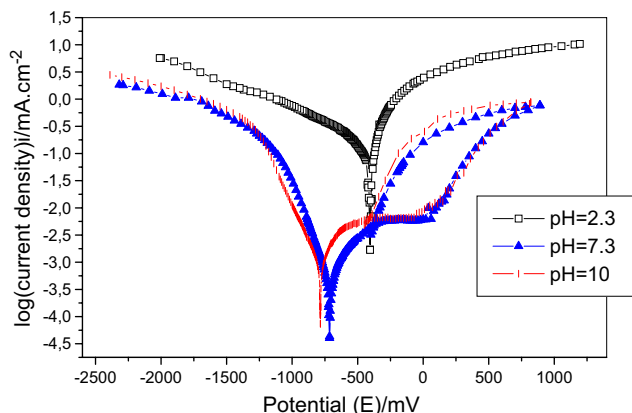$$Al(OH)_3 + 3H^+ \leftrightarrow Al^{3+} + 3H_2O$$
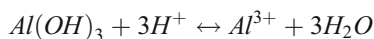
This reactional delay justifies the discrepancy in b_c. It can't be interpret in terms of diffusion where no flat appears in the final part of the curve.

The anodic processes are more dependant on pH than the cathodic ones, because here the profiles are also different between the acid medium and the two others. As a matter of fact, an acid attack is increasing continuously, which means that the dissolution of aluminium is always running, according to the reaction:

$$Al \rightarrow Al^{3+} + 3e^-$$
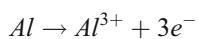
The latter decomposes indeed [10, 11] into a monoelectronic determining slow step and bi-electronic fast step.

While in the other mediums, a passivating steady-state [2] is observed.

Table 2 below indicates passivation parameters close to each other for the neutral and alkaline solutions. Though slightly different, the E_{rup} values followed by identical transpassive dissolution (therefore independent on the pH), were found by Carroll and Breslin [12] in pure Al and concentrated NaCl however. The passive film is consequent to the reaction between dissolved Al^{+3} and the OH$^-$ of the solvent, strengthening thus the initial hydroxide. The pH values measured after each polarisation indicate constancy in this parameter in the acid medium, but a slight decrease in the neutral solution, and more important in the alkaline one where the hydroxide film is consummated by the OH$^-$ ions according to the reaction:

$$Al(OH)_3 + OH^- \leftrightarrow Al(OH)_4^-$$

These are also conclusions of some authors [13].

The anodic slopes b_a, very high in neutral and alkaline mediums, are justified by the occurrence of the passivation steady-state and specific adsorption phenomena dealing with the presence of OH$^-$ and Cl$^-$ ions. Besides, with pH= 2.3, the closer value to that theoretical of a pure transfer expresses very well an active and increasing or a little delayed process.

The behaviour of Al-12Si through the electrokinetic parameters linked with corrosion (i_{corr}, R_P and P) is very influenced by the pH, harmful acid and the most favourable neutral medium. A perfect harmony exists between the two methods for establishing i_{corr} or when these values are compared to R_P and P. They mean the validity of the Stern

Fig. 5 Effect of the pH on the electrochemical behaviour of the Al-12Si alloy polarized in NaCl 10^{-3} M

Table 2 Electrokinetic parameters determined from total polarization curves of the Al-12Si/NaCl system, at different pH

Electrokinetic parameters	PH=2.3	pH=7.3	pH=10
E_{corr}(mV/ECS)	−405	−715	−785
$-b_c$(mV/dec)	320.51	144.09	184.16
b_a(mV/dec)	108.89	225.73	254.45
i_{corr}(graphic) ($\mu A.cm^{-2}$)	89.12	0.44	0.92
i_{corr}(S-G) ($\mu A.cm^{-2}$)	84.06	0.37	0.99
R_p($\Omega.cm^2$)	0.58	103.95	46.73
$P(10^3\ cm)$	990.26	4.91	10.22
i_{pass}($\mu A.cm^{-2}$)	–	5.81	6.47
L_{pass}(mV)	–	340	364
E_{rup}(mV/ECS)	–	19.86	−66.32
E_{rep}(mV/ECS)	–	−368.84	−401.41
pH before polarisation	2.3	7.1	10
pH after anodic polarisation	2.5	6	7.2
pH after cathodic polarisation	2.3	6.5	7.9

and Geary relation and of the approximation near equilibrium for the small over voltages and the suitable determination of the Tafel slopes.

Now, we will focus our attention on the nature of the electrolyte. The curves reflecting the influence of the addition of Cu^{2+}, Zn^{2+}, SO_4^{2-}, NO_3^- and PO_4^{3-} in comparison with NaCl 10^{-3} M are illustrated in Fig. 6. Their exploitation allowed the access to passivation and electrokinetic parameters which are shown in Table 3 below.

All the cathodic portions show more or less increasing profiles, without any deviation. So, it is a monoelectronic reduction of protons since the initial pH which is slightly acid becomes neutral, if they do not stay constant (like in the case of NO_3^- and PO_4^{3-}). The presence of a wave due to a second simultaneous reaction of metallic deposition like with Cu^{2+} or Zn^{2+} was not produced because of the low concentration used. All left-hand side curves with additional ions are close to that of NaCl alone. Consequently, the modification brought out by these ions in the kinetic process of the system is negligible. The $|b_c|$ value belonging to NaCl alone (185 mV/dec) has been reduced by the additional ions, since $|b_c|$ is in the range 120/140, justifying thus a pure activation process with no or a small delay.

The anodic portions offer an obvious scattering after the incorporation of the considered ions. This is to say that such anodic process is sensitive to the nature of the electrolyte. Indeed, if NaCl alone shows a slight shouldering in the evolution of the current, in revenge Cu^{2+}, Zn^{2+}, SO_4^{2-} and NO_3^- and at a lesser degree PO_4^{3-} present passivating steady-states which are not very protecting however. In all cases, these ions slow down the dissolution of the alloy, relative to NaCl alone (anodic currents weaker for the same over voltage).

Thus the occurring of a superficial film does not always mean a protection against pits, according to the conclusions drawn up by Aogaki [14]. This steady-state could rather be attributed to the adsorption of the considered ions at the surface of the alloy, and that does not prevent the current from causing damages [15]. Besides, with Cu^{2+} and Zn^{2+}, fluctuations are registered over the breaking potential E_{rup}. They are justified by the heterogeneity of the surface, generated by the spontaneous deposition of copper and zinc. Conversely to b_c, the b_a values have all been increased by the addition of the ions studied. They express a larger delay with comparison to NaCl alone. This delay is explained by the passivation to overcome, the spontaneous depositions to avoid and the adsorption to eliminate, in between other reasons.

The electrokinetic parameters show a better behaviour of the alloy when the considered metallic ions are added (R_p increases and i_{corr} decreases). No amelioration with SO_4^{2-} and PO_4^{3-} with respect to NaCl alone (parameters nearly constant) and a worse behaviour in the presence of NO_3^-. However, the effect of the nature of the ions is less than that of the pH. A satisfactory correlation is registered between all these parameters for the cations, confirming thus the linearity of $i=f(E)$ near equilibrium and the validity of the Stern and Geary law. However for the anions, a discrepancy exists (ratio 2) between the graphical i_{corr} and the calculated one. To settle, one refers to R_p, a trustworthy parameter which is based on the linearity between E and i, and where the electrochemical system remained close to its stable initial state. Now R_p has been reduced (from 52 to 37), therefore calculated i_{corr} seems more correct. Indeed, for graphical ones, there are uncertainties arising from the determination of Tafel slopes. This is what happens also with SO_4^{2-} and PO_4^{3-}.

Sato [16] reported that in the case of iron, the adsorption of SO_4^{2-} intensifies the oxide layer and hinders the ions transfer
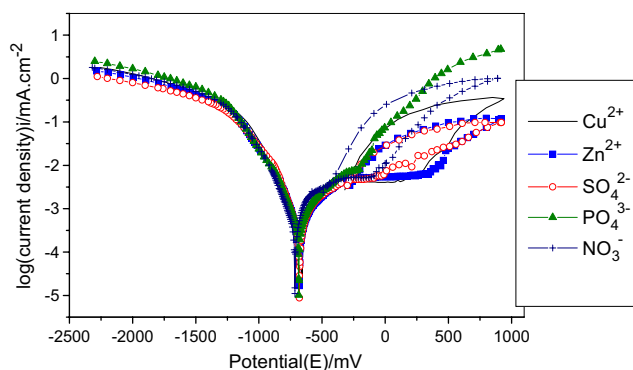

Fig. 6 Influence of the addition of Cu^{2+}, Zn^{2+}, SO_4^{2-}, NO_3^- and PO_4^{3-} (10^{-6} M) to NaCl (10^{-3} M) on the Al-12Si alloy. Total polarization curves

Table 3 Passivation and electrokinetic parameters for Al-12Si/NaCl 10^{-3} M deduced from the polarization curves, when Cu^{2+}, Zn^{2+}, SO_4^{2-}, NO_3^- and PO_4^{3-} (10^{-6} M) are incorporated

Electrokinetic Parameters	NaCl 10^{-3} M	NaCl 10^{-3} M+ CuCl$_2$ 10^{-6} M	NaCl 10^{-3} M+ ZnCl$_2$ 10^{-6} M	NaCl 10^{-3} M+ Na$_2$SO$_4$ 10^{-6} M	NaCl 10^{-3} M+ NaNO$_3$ 10^{-6} M	NaCl 10^{-3} M+ Na$_3$PO$_4$ 10^{-6} M
E_{corr}(mV/ECS)	−715	−660	−680	−680	−715	−685
$-b_c$(mV/dec)	185.53	143.88	127.06	123.61	131.4	134.95
b_a(mV/dec)	183.15	248.14	222.22	214.59	206.66	224.72
i_{corr}(graphic) (μA.cm^{-2})	0.62	0.36	0.32	0.32	0.43	0.39
i_{corr}(S-G) (μA.cm^{-2})	0.76	0.47	0.45	0.61	0.94	0.68
R_p(Ωcm^{-2})	52.35	83.33	78.12	55.55	37.04	53.76
P(10^3 cm)	6.87	4.02	3.59	6.62	4.75	4.34
i_{pass}(μA.cm^{-2})	15.06	4.22	5.10	(4.71)	5.01	7
L_{pass}(mV)	300	360	644.5	(240)	235	88
E_{rup}(mV/ECS)	−108.22	79.43	344.52	(−30)	−94.82	−216.94
E_{rep}(mV/ECS)	–	−297.36	−248.21	(−272.04)	−453.37	–
pH before polarisation	6.05	6.05	6.4	6.4	6.8	5.8
pH after anodic polarisation	6.8	6.5	6.4	6.5	6.25	5.7
pH after cathodic polarisation	6.95	6.4	6.8	6.6	6.35	5.8

and therefore the corrosion. With Al-12Si, this effect is not confirmed, already because of it is at weak concentration.

Conclusion

The electrokinetic behaviour of Al-12Si is dependent on the reagent concentration:

The reduction process in the electrolytes with mixed halides increases with the concentration of I$^-$ or Cl$^-$ and goes in the way of consumed protons or produced hydroxides (finale pH higher than the initial one). The cathodic rate is favoured by an increasing conductivity when the concentration increases. This reaction is governed by a pure transfer.

The oxidation currents depend on the Cl$^-$ concentration, but not on the I$^-$ one. The difference is probably due to the properties of each ion (size, energy...). The films produced are fragile because they are instable and this is due to adsorptions: distortions in potentials and a reaction delay which remove b_a from its theoretical value because of the repassivation phenomenon.

With only one halide, the alloy offers a resistance ten times better in Cl$^-$ than I$^-$, once these two anions are put together, the behaviour becomes similar.

The electrokinetic performances of the alloy considered are more dependent on the pH: The process rates, the profiles and mechanisms, mainly the anodic ones, vary with the pH. Besides, the b_c value in the acid medium implies a pure transfer delayed by a chemical secondary reaction, relative to the other mediums, and conversely to the anodic process where the acid solution offers an active and less delayed mechanism.

The behaviour of Al-12Si with respect to corrosion depends on the solution nature: If the cathodic portions reflect a similar behaviour for the incorporated ions with close profiles, in revenge, the anodic portions are more scattered with variable passivating flats and not very resistant as also fluctuations (micro piles or local corrosion) registered with Cu^{2+}, Zn^{2+} and SO_4^{2-}.

The incorporated cations confer to the alloy immersed in NaCl, a better resistance with regard to corrosion, in comparison with the anions and mainly NO_3^-.

In general, and despite of tricky experiences characterised by the aggressive nature of ions, negligible concentrations and aerated mediums which increase the instability of systems, reproducible results have been established. Except for anions, correlations between the two i_{corr}, and between i_{corr} and R_p are very satisfactory, confirming thus on the one hand the validity of the Stern and Geary law, the linearity near equilibrium of i=f(E) (Ohm type law), and on the other hand, the correct localisation of the Tafel area, and the determination of the parameters independently of the studying range.

By way of perspectives, these investigations could be extended to combinations of these or other ions, in a larger range of concentration and pH and to other variables such as the amount of O$_2$ and the surface treatment.

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