

# The effect of chloride concentration and pH on pitting corrosion of AA7075 aluminum alloy coated with phenyltrimethoxysilane

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**Abstract** The effect of chloride ion concentration and pH of solution on the corrosion behavior of aluminum alloy AA7075 coated with phenyltrimethoxysilane (PTMS) immersed in aqueous solutions of NaCl is reported. Potentiodynamic polarization, linear polarization, open circuit potential, and weight loss measurements were performed. The surface of samples was examined using SEM and optical microscopy. Elemental characterization of the coating by secondary ion mass spectrometry indicates an intermediate layer between coating and aluminum alloy surface. The corrosion behavior of the aluminum alloy AA7075 depends on chloride concentration and pH of solution. In acidic or neutral solutions, general and pitting corrosion occur simultaneously. On the contrary, exposure to alkaline solutions results in general corrosion only. Results further reveal that aluminum alloy AA7075 is susceptible to pitting corrosion in all chloride solutions with concentrations between 0.05 M and 2 M NaCl; an increase in the chloride concentration slightly shifted both the pitting and corrosion potentials to more active values. Linear polarization resistance measurements show a substantially improved corrosion resistance value in case of samples coated with PTMS as compared to uncoated samples in

both neutral (pH=7), acidic (pH=0.85 and 3), and alkaline chloride solutions (pH=10 and 12.85). The higher corrosion resistance of the aluminum alloy coated with PTMS can be attributed to the hydrophobic coating which acts as a barrier and prevents chloride ion penetration and subsequent reaction with the aluminum alloy.

**Keywords** Aluminum alloy (AA7075) · Phenyltrimethoxysilane · Pitting corrosion · Sodium chloride · Harrison's solution

## Introduction

Aluminum and its alloys are generally passive and corrosion resistant in aqueous solutions [1] except for pitting corrosion caused by reactive species, such as halide ions (e.g., chloride) [2–5]. The passive film on the aluminum alloy surface is a poor electronic conductor; cathodic reactions occur on micron-sized particles of impurity constituents or small precipitate particles [6–18]. Various grades of aluminum alloy and metals were extensively used to study the effect of alloying elements on the breakdown of the passive film [6–15]. The presence of alloying elements in the microstructure such as insoluble intermetallic particles ( $\text{Al}_2\text{Cu}$ ,  $\text{FeAl}_3$ ) or single elements (Cu, Si) leads to formation of local electrochemical cells between them and the aluminum matrix [6–23]. This causes highly localized attack by pitting in aggressive medium [2–5, 24, 25]. Various factors affect the pitting corrosion in aluminum alloys such as the type of aggressive ion and its concentration, the pH of the media, the temperature, or the structural characteristics of the oxide passive film [5–26]. As an efficient replacement of highly toxic chromium-containing corrosion protection coatings, thin layers of

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various materials obtained via sol–gel processing have been proposed and evaluated [27, 28]. In the present work, we prepared aqueous phenyltrimethoxysilane (PTMS) sol–gel coatings on aluminum alloy AA7075 for corrosion protection and characterized the corrosion resistance of coated aluminum alloy by potentiodynamic polarization test and electrochemical methods. We have examined the effect of the concentration of  $\text{Cl}^-$  ions and pH of electrolyte solutions on the localized corrosion of both uncoated and PTMS-coated samples of aluminum alloy AA7075.

## Experimental

### Materials

PTMS (Alfa Aesar, 97%), acetic acid (Fluka, 99%, used as a catalyst), acetylacetone (Sigma-Aldrich, 99%, used to moderate the reaction speed), and *n*-propanol (Fisher, 99.9%, used as cosolvent to prevent precipitation) were used as received.

### Preparation of the samples

The aluminum alloy (AA7075) was cut into discs (3 cm diameter, 0.5 cm thickness) and polished with a Beta grinder/polishing machine with SiC abrasive paper grit 600. Samples were cleaned and degreased ultrasonically with ethanol and distilled water for a few minutes. Finally, they were cleaned with ethanol and dried with air prior to the spin-coating process. The precursor solution was applied onto the substrates from a syringe. The spinning speed was set to 4,000 rpm for 90 s.

### Solutions

We prepared electrolyte solutions with different concentrations of NaCl (pro analysis (p.a.) grade, Merck, 0.05, 0.5, 1, 1.5, and 2 M) with distilled water and analytical grade reagents. The pH values (0.85, 3, 7, 10, and 12.85) were adjusted by adding NaOH (p.a., Merck) or sulfuric acid (95–97%, p.a., Merck). Harrison's solution was prepared by dissolving 3.5 g/l of  $(\text{NH}_4)_2\text{SO}_4 + 0.5$  g/l NaCl in distilled water. Solutions were not purged with inert gas but exposed to ambient air.

### Coating

PTMS was used as an alkoxide precursor for sol–gel process. PTMS (2.5 ml), 1.25 ml acetic acid, and 1.25 ml of acetylacetone were placed in a round-bottom 250-ml flask. The solution was stirred for 5 min at ambient temperature. A mixture of distilled water (0.5 ml) and *n*-propanol (2 ml) was added to the solution and stirred for 90 min to complete

hydrolysis and condensation reactions. The resulting solution was deposited on the polished aluminum alloy samples with spin-coating at 4,000 rpm for 90 s. Finally, the samples were heated at 573 K for 150 min in a furnace.

### Weight loss measurements

Tests were done in 250-ml glass beakers containing 150 ml of 0.05 M NaCl solution with different pH values at ambient temperature. The samples were weighed before introduction into the test solutions. To determine the loss of weight, the samples were removed after immersion for various numbers of days, washed with distilled water, dried, and then weighed again [29, 30]. Corrosion rate was determined according to  $\text{CR} = 3.45 \cdot 10^6 \cdot w / (adt)$  with  $w$  = weight loss in grams,  $a$  = exposed surface area in square centimeters,  $d$  = density of the sample in grams per cubic centimeter, and  $t$  = time of exposure in hours.

### Potentiodynamic polarization

Electrochemical measurements were carried out in a conventional three-electrode cell with the aluminum samples (3 cm diameter and 0.3 cm thickness) as working electrode, a Ag/AgCl reference electrode (saturated with KCl), and a platinum counter electrode. Cyclic potentiodynamic current vs. potential curves were recorded in the range of  $-1.5 > E_{\text{Ag/AgCl}} > 0$  V at  $dE/dt = 50$  mV  $\cdot$  s $^{-1}$  at ambient temperature. The electrolyte solution contained 0.05 M NaCl at different pH values or different concentrations of NaCl at pH=7. A Princeton Applied Research Potentiostat (Parstat 2273) was used with Power Suite 2.58 software. The exposed surface area was 2 cm $^2$ .

### Porosity measurements

Potential scans with a Princeton Applied Research potentiostat Parstat 2273 and Power Suite 2.58 Software were used to measure the film porosity electrochemically. The samples were used as working electrode in a three-electrode setup with a platinum counter electrode and a saturated AgCl reference electrode. The dissolution current density of the samples was measured in the potential range of  $-1.5 > E_{\text{Ag/AgCl}} > 0$  V at  $dE/dt = 50$  mV s $^{-1}$  at ambient temperature. A mildly corrosive electrolyte solution and a relatively fast scan rate were used in order to minimize corrosive damage to the sample. Under these conditions, the current density  $j_{\text{corr}}$  at  $E = E_{\text{OCP}} + 50$  mV chosen for the porosity evaluation depends primarily on the area of the exposed, uncoated metal. The average film porosity can be calculated from the ratio of the maximum dissolution current densities of coated and uncoated reference samples [31]. This method has been successfully applied with

different coating systems before and is described in detail elsewhere [32].

Open circuit potential measurements

Open circuit potential (OCP) was recorded during 10 min in electrolyte solutions of various pH values (0.85, 3, 7, 10, and 12.85).

Linear polarization resistance measurements

Most electrochemical corrosion test experiments measure a resistance that is representative of the rate of the corrosion reaction. This is true for linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS) techniques. These resistances are related to the polarization resistance by the Stern–Geary linear approximation of the Butler–Volmer equation (1)

$$R_p = B/I_{corr} \tag{1}$$

with the corrosion current  $I_{corr}$  and the Stern–Geary constant  $B$  calculated according to

$$B = B_a \cdot B_{ca}/2.303(B_a + B_{ca}) \tag{2}$$

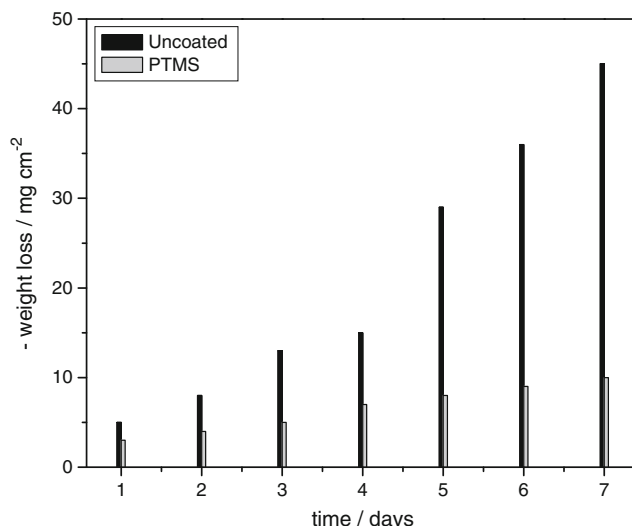
using the Tafel slopes  $B_a$  and  $B_{ca}$  of the anodic and cathodic parts of Tafel plots in millivolts per decade. Linear polarization measurements were performed at  $dE/dt = 2.0 \text{ mV}\cdot\text{s}^{-1}$  with the samples previously immersed for 24 h in 0.05 M NaCl solution. Corrosion inhibition efficiency was calculated according to  $IE (\text{percentage}) = [R_{p,co.} - R_{p,un.}/R_{p,co.}] \cdot 100$  [33].

Electrochemical impedance spectroscopy

EIS was used to evaluate the electrochemical behavior of the coated samples in 0.05 M NaCl solution at OCP at ambient temperature. A conventional three-electrode cell with the sample as working electrode, a platinum counter electrode, and Ag/Cl (saturated with KCl) reference electrode was used. The exposed surface area was  $2 \text{ cm}^2$ . The data were obtained using a sine wave of 10 mV

**Table 1** Weight loss of uncoated and coated samples of alloy AA7075 at different pH values after 1 day of immersion in a solution of 0.05 M NaCl

pH	Weight loss/(mg cm <sup>-2</sup> )	
	Uncoated	PTMS
0.85	307	6
3	13	4
7	10	1
10	14	4
12.85	555	12

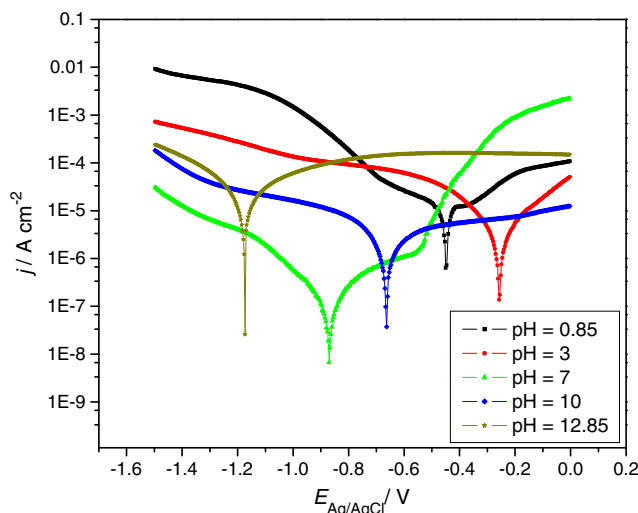


**Fig. 1** Weight loss as a function of immersion time in a solution of 0.05 M NaCl at pH 7

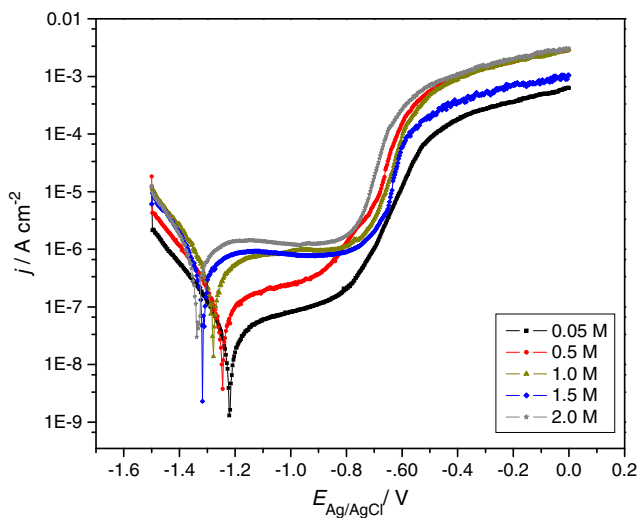
amplitude between 100 kHz and 39.8 mHz with ten points in every frequency decade.

Structural characterization

Secondary ion mass spectrometry (SIMS) was used to obtain information about the element distribution within the coating layer using a double focusing sector field instrument (Cameca ims 5f) with 17 kV O<sup>-</sup> primary ions. As the layer also contains oxygen, the use of cesium primary ions would have been advantageous, but suffered from sample charging effects, even with an additional gold coating on top. Sputtered positive secondary ions were detected from the inner 60 μm of an area of 150·150 μm<sup>2</sup>.



**Fig. 2** Polarization curves for samples of AA7075 coated with PTMS at different pH values after immersion for 1 day



**Fig. 3** Anodic polarization curves obtained with samples of uncoated AA7075 in solutions of different concentrations of NaCl at pH=7

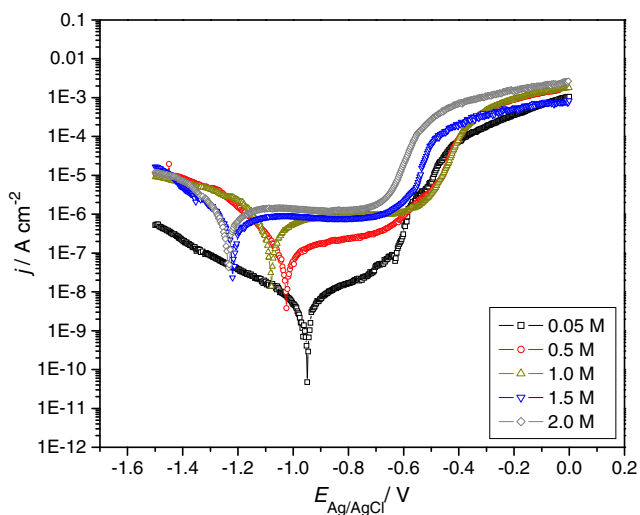
### Surface analysis

#### Scanning electron microscopy

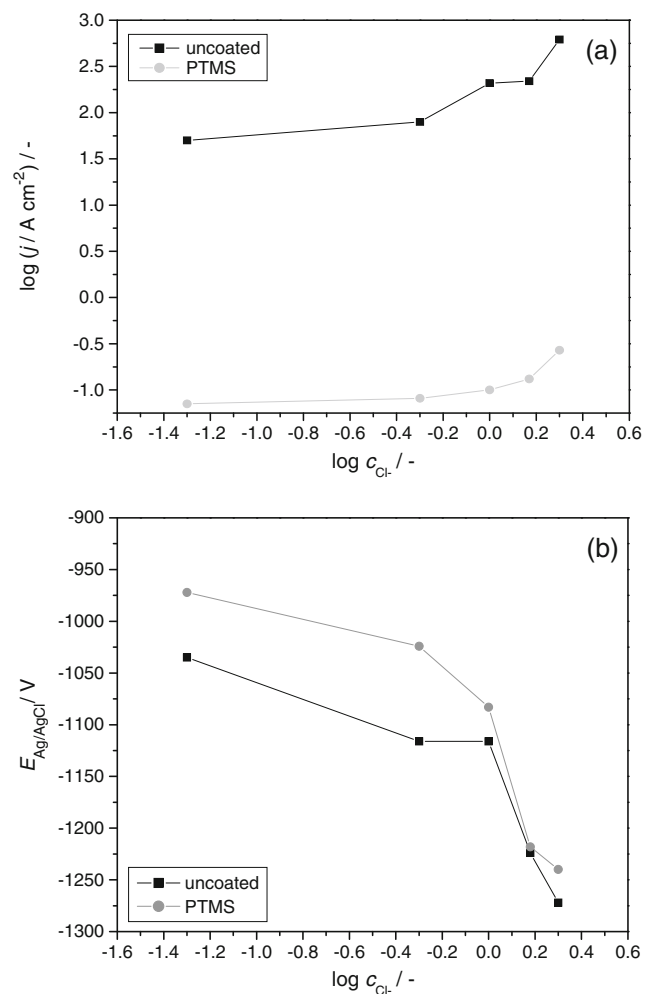
Scanning electron microscopy (SEM) images were obtained using a digital scanning electron microscope Philips XL30-FEG at 100  $\mu\text{A}$ .

#### Secondary ion mass spectrometry

Depth profiles of coated samples were obtained using a Cameca ims5f instrument.



**Fig. 4** Anodic polarization curves obtained with samples of AA7075 coated with PTMS after immersion in solutions of different concentrations of NaCl at pH=7



**Fig. 5** Dependency of corrosion current density  $j_{\text{corr}}$  (a) and corrosion potential  $E_{\text{corr}}$  (b) for uncoated and coated samples of AA7075 on the concentration of  $\text{Cl}^-$  ion

### Optical microscopy

After immersion of the samples in neutral electrolyte solution (0.05 M NaCl and Harrison's solution) for 7 days, the samples of Al alloy were examined using an optical microscope Model Olympus BX 50 with camera Nikon DXM 1200.

## Results and discussion

### Weight loss experiments

The weight loss of uncoated and coated samples stored in aqueous solution 0.05 M NaCl at different pH values (0.85, 3, 7, 10, and 12.85) at ambient temperature as obtained from the difference between weight of the samples before and after immersion for 1 day is shown in Table 1. In neutral solution, the decrease in weight loss caused by the

**Table 2** Linear polarization results of uncoated and coated specimens after immersion in electrolyte solutions with different pH values for 10 min

pH	Uncoated				PTMS				
	$E_{corr}$ /mV	$E_{pitt}$ /mV	$j_{corr}$ / $\mu\text{A}\cdot\text{cm}^{-2}$	$R_p\cdot 10^3$ / $\Omega\cdot\text{cm}^2$	$E_{corr}$ /mV	$E_{pitt}$ /mV	$j_{corr}$ / $\mu\text{A}\cdot\text{cm}^{-2}$	Porosity /%	$R_p\cdot 10^3$ / $\Omega\cdot\text{cm}^2$
0.85	-621	-552	2,010	0.06	-369	-294	4.0	0.2	1
3	-336	-278	50.7	1.38	-261	-192	4.6	9.1	27
7	-1,010	-600	34.4	1.68	-870	-555	0.18	0.5	5,214
10	-1,040	-591	36.5	1.36	-663	-576	1.87	5.1	600
12.85	-1,340	-1,269	1560	0.01	-1,212	-1,095	46.3	4.5	2

coating is relatively smallest, whereas in acidic (pH=0.85 and 3) or alkaline (pH=10 and 12.85) media, the inhibition effect is significantly larger. In a second run with neutral 0.05 M NaCl, weight losses as a function of time of immersion as shown in Fig. 1 were obtained. Again, no complete corrosion inhibition is afforded, but coating with PTMS resulted in significant corrosion protection.

Potentiodynamic test

Potentiodynamic polarization curves for AA7075 samples coated with PTMS obtained in solutions of 0.05 M NaCl at pH values of 0.85, 3, 7, 10, and 12.85 with distinctly different shapes are shown in Fig. 2. The corrosion current density in neutral solution is the lowest. At constant pH=7 both with uncoated (Fig. 3) and coated samples (Fig. 4), a change of the concentration of NaCl from  $c=0.05$  to 2.0 M leads to decreasing corrosion potential ( $E_{corr}$ ) and increasing current density ( $j_{corr}$ ) values. Figure 5a, b shows the dependence of  $j_{corr}$  and  $E_{corr}$  on concentration of NaCl. Relationships between corrosion current density  $j_{corr}$  and corrosion potential  $E_{corr}$  upon chloride ion concentration and hydrostatic pressure have been examined elsewhere [34], and they are based on a shift of the reaction equilibrium between aluminum and chloride ions on one hand and chloride-covered aluminum surface (and an electron released during this process) on the other hand. Increase of chloride concentration results in a shift of the equilibrium and concomitant changes of  $j_{corr}$  and  $E_{corr}$ . The linear relationship in case of  $E_{corr}$  can be represented by the equation:

$$E_{corr} = E_{corr}^0 + 2.303((R \cdot T)/(n \cdot F))\log c \tag{3}$$

where  $E_{corr}^0$  is the corrosion potential at unit electrolyte concentration and  $c$ , concentration of chloride ions. The non-unit value of  $n$  (as expected for a single-electron transfer) may be related to a multistep process of corrosion product formation. The dependence of  $E_{corr}$  on concentration is smaller in the case of samples coated with PTMS than with uncoated samples of aluminum alloy AA7075 because coating makes major parts of the metal surface inaccessible for chloride adsorption; the reaction equilibrium addressed above is less affected.

The porosity (expressed as percentage of uncoated electrode surface area) of the coating is calculated from the values of dissolution current density  $j_{corr}$  at  $E=E_{OCP}+50\text{ mV}^1$  (see Table 2) for uncoated and coated samples according to:  $\text{porosity} = j_{corr,coat} / j_{corr,uncoat} \cdot 100/\text{percentage}$ . Results are summarized in Table 2. Corrosion currents of the coated samples are significantly lower obviously because most of the surface is covered with a protective coating, and the porosity also shows some dependency on the pH value of the electrolyte solution. Chemical reactions between solution constituents may be responsible for changes in porosity, and details of this interaction showing somewhat unexpectedly highest porosity not at an extreme pH value are not yet resolved.

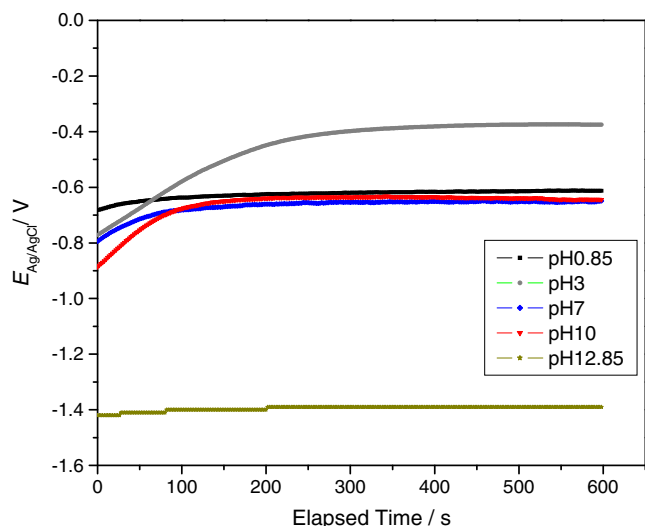
It was found that OCP becomes more positive with time at all tested pH values in case of uncoated and coated samples (Figs. 6 and 7, respectively) as previously observed by Milosev et al. [35]. OCP becomes more negative with increasing pH values. The OCP curves of coated and uncoated AA7075 samples exposed to solution of pH=0.85, 3, 7, 10, and 12.85 are different in values and shape. Both with uncoated and coated samples in strongly acidic and alkaline solutions, a stable corrosion potential is rapidly attained because of the limited stability of any passivating layer. With less extreme pH values, formation of a passive layer on both samples needs longer times finally resulting in a more positive corrosion potential implying increased passivity. In the case of the coated alloy sample, the process is particularly poorly defined, resulting in a somewhat unstable corrosion potential. Potential oscillations observed are due to dissolution/repassivation phenomena.

Linear polarization resistance

LPR values of uncoated and coated samples in electrolyte solutions of NaCl with different pH values (0.85, 3, 7, 10, and 1.85) are shown in Table 2. The Tafel extrapolation method [5] was used to calculate the corrosion potential ( $E_{corr}$ ), pitting potential ( $E_{pitt}$ ), and corrosion current density ( $j_{corr}$ ). According to Table 2 and Fig. 8, with increasing pH value, the

<sup>1</sup> For position of this potential see Figs. 3 and 4.



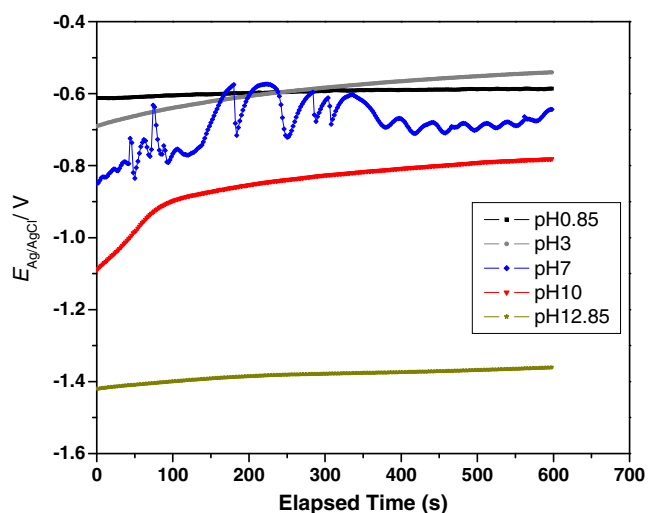


**Fig. 6** Open circuit potential vs. time of immersion of uncoated aluminum alloy AA7075 in solutions of 0.05 M NaCl at various pH values

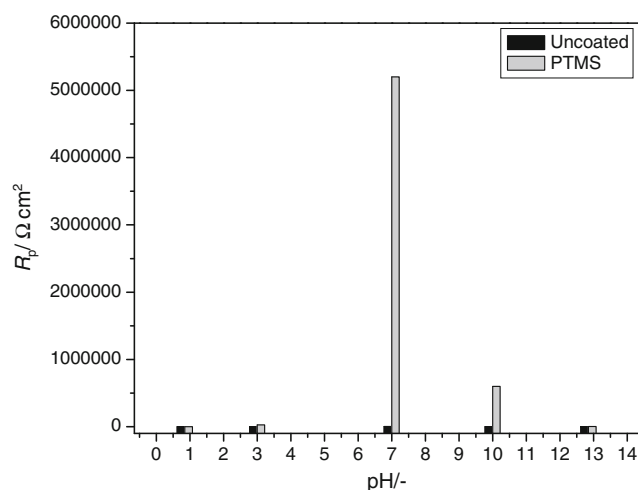
uncoated sample shows an increase of  $j_{\text{corr}}$  (corresponding to the decrease  $R_p$ ) reaching the highest value at pH=7; beyond this value, the change goes into the opposite direction. Values are lowest in acidic and basic media. The values of  $E_{\text{corr}}$  increase with pH continuously. The results of coated samples with PTMS demonstrate, that the values of  $j_{\text{corr}}$  record lowest values compared to uncoated, and  $R_p$  record highest values compared with uncoated samples.

#### Electrochemical impedance spectroscopy

From impedance measurements, the corrosion resistance of uncoated and coated samples was obtained. Coating caused an increase of the resistance from  $1 \cdot 10^3$  to  $159 \cdot 10^3 \Omega \text{ cm}^2$

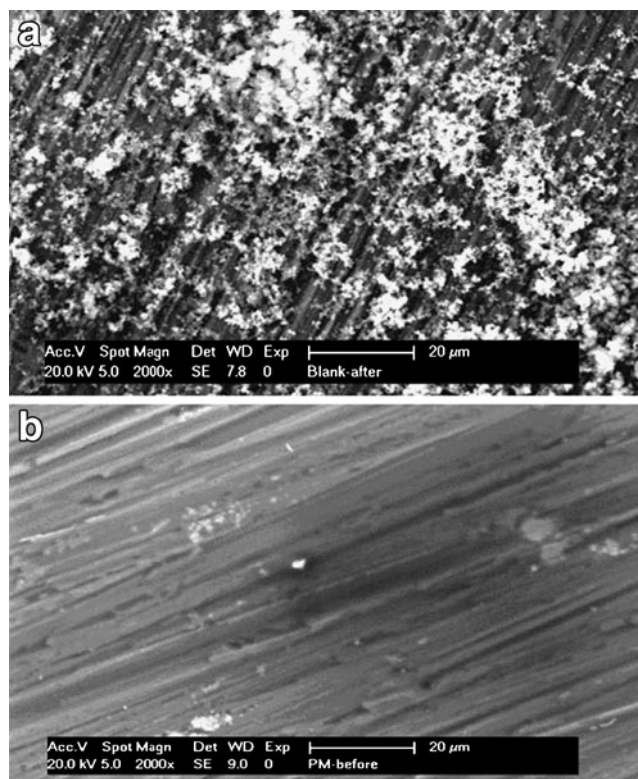


**Fig. 7** Open circuit potential vs. time of immersion of aluminum alloy AA7075 coated with PTMS in solutions of 0.05 M NaCl at various pH values

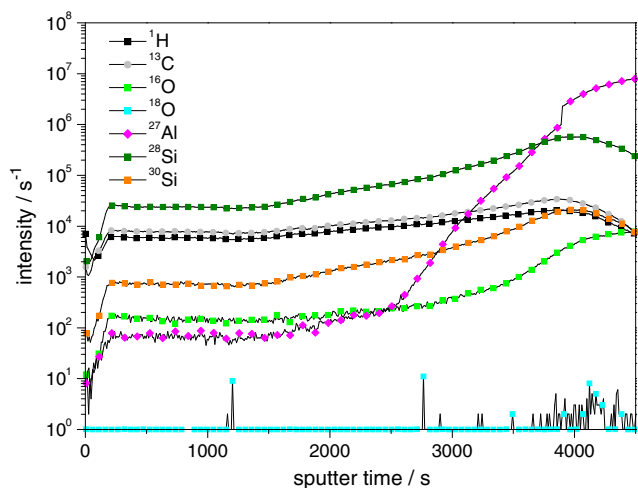


**Fig. 8** Variation of polarization resistance of aluminum alloy AA7075 in 0.05 M NaCl solution as a function of pH value for uncoated and coated samples

as measured after 1 day of immersion in 0.05 M NaCl solution, further confirming the corrosion-protection capability of the PTMS-based silica sol-gel coating. The calculated corrosion current density decreased from  $j_{\text{corr}} = 14$  to  $0.14 \mu\text{A cm}^{-2}$ ; this is equivalent to a corrosion inhibition efficiency of 99%.



**Fig. 9** Scanning electron micrograph of uncoated (a) and coated (b) samples of AA7075 after 1 day immersion in a solution of 0.05 M NaCl



**Fig. 10** Depth profiles of AA7075 aluminum alloy coated with PTMS and annealed 573 K

SEM

SEM of the corroded surfaces of uncoated (a) and coated samples AA7075 after immersion in 0.05 M NaCl solution for 1 day at neutral pH are shown in Fig. 9a and b, respectively. The coating had a dense structure with a uniform distribution of hillocks on the surface. We believe that these hillocks on the surface coating are due to self-

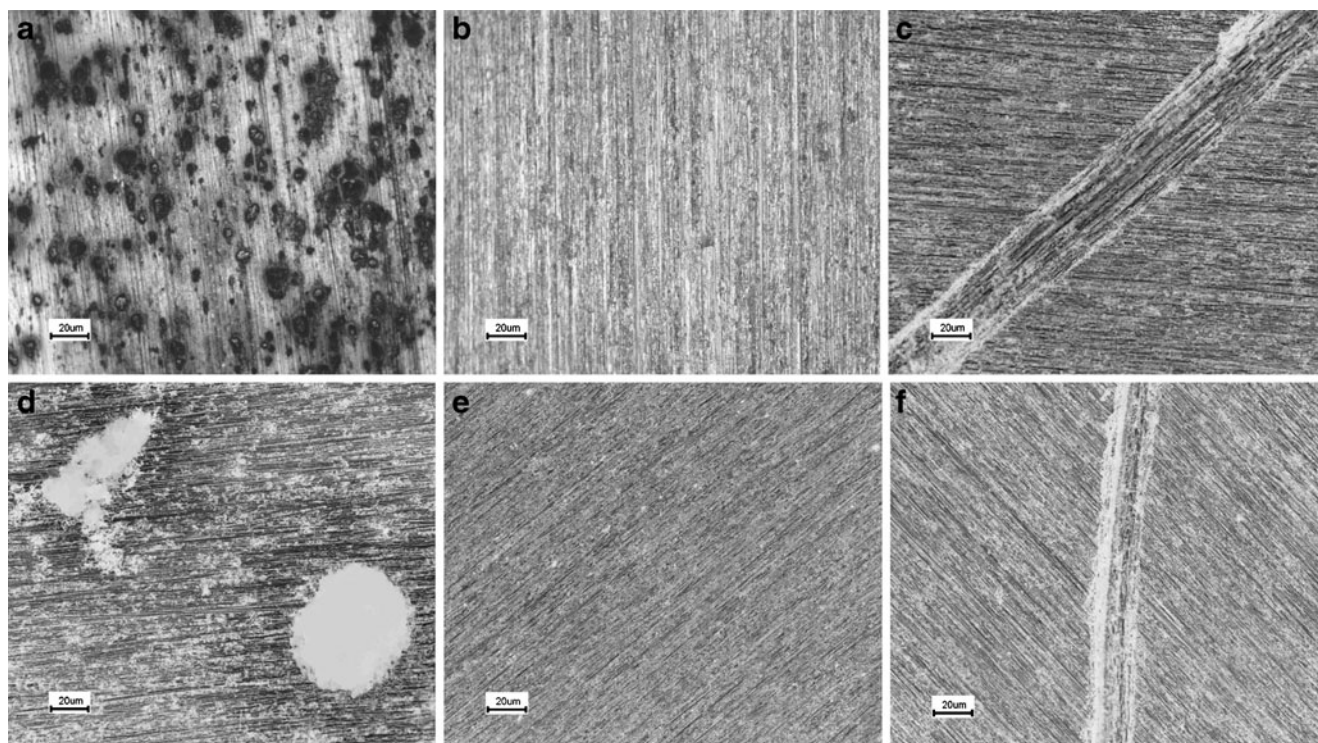
assembly of the macromolecular structures during the hardening process. There is no sign of cavities, pin holes, or any other type of surface defects. The thickness of the coating layer was approximately 89 nm as estimated with a Dektak 8000 instrument (not shown here).

SIMS

Figure 10 shows a depth profile of the coating annealed at 573 K. The coating contains, as expected, Si, H, C, and O, but also Al. The slow rise of the signal due to aluminum starting at about 2,700 s sputtering time implies the presence of a mixed intermediate layer containing both metal and species from the sol-gel coating. This may be caused by a chemical reaction between the aluminum surface and the slightly acidic coating solution. The overall signal growth may be due to a more dense coating at the bottom.

Optical microscopy

Figure 11 shows optical micrograph of the film formed upon the surface of coated and uncoated samples after immersion for 7 days in different electrolyte solutions (0.05 M NaCl and Harrison's solution). Hemispherical pits with different sizes were grown throughout the surface of



**Fig. 11** Optical microscope picture of uncoated sample after 7 days of immersion in 0.05 M NaCl (a) and Harrison's solution (d) at pH=7; coated samples after 7 days of immersion in 0.05 M NaCl (b) and

Harrison's solution (e); scratched sample coated with PTMS corroded in the scratch area after immersion in 0.05 M NaCl (c) and Harrison's solution (f) for 7 days

the samples, and around each pit, a white corrosion product or white gelatinous mass was accumulated as the result of the corrosion of the aluminum alloy matrix in the vicinity of the precipitates. These micrographs clearly show that the damage caused by this type of corrosion is accentuated with uncoated samples exposed to neutral solution.

## Conclusions

Treatment of aluminum alloy AA7075 with PTMS coating offers a good corrosion protection at different concentrations of NaCl. Immersion of uncoated and samples coated with PTMS for 1 day at different pH values and for 7 days in neutral solution of 0.05 M NaCl proves that PTMS can be used as an excellent coating to prevent aluminum alloy AA7075 from corrosion. The concentration of chloride ions plays an important role in the rate of corrosion: The current density increases with concentration. The OCP for uncoated and coated samples depends on pH value; the value decreases when pH decreases.

PTMS provides a coating effective at all pH values, in particular, at low and high values where naturally formed passivation layers are not effective. The coating is also effective at all chloride concentrations, in particular again, at high concentration values; the sample applies to the change of  $E_{\text{corr}}$ . The corrosion protection performance of the PTMS-treated samples is very promising as an alternative to toxic chromate coating.

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