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► **To cite this version:**

M.M. Pricopi, R. Chelariu, N. Apostolescu, D.-M. Gordin, D. Sutiman, et al.. Corrosion behavior of some timonb alloys in ringer solution. *Revista de Chimie -Bucharest- Original Edition*, 2020, 71 (7), pp.187-196. 10.37358/RC.20.7.8236 . hal-02960239

HAL Id: hal-02960239

<https://hal.science/hal-02960239>

Submitted on 12 Oct 2020

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Corrosion Behavior of Some TiMoNb Alloys in Ringer Solution

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Abstract. *The aim of this study was to investigate the influence of different process parameters as chemical composition, the pH value and immersion time on the corrosion of the some TiMoNb alloys, using different electrochemical techniques such as: cyclic voltammetry, open circuit potential (OCP) measurement, polarization curves and electrochemical impedance spectroscopy (EIS). The alloys were analyzed in the natural pH of the Ringer solution, but also with an acidic modification of the solution (ph = 4) and a basic modification (ph = 8). The more acidic values of pH, the more evident are differences between corrosion behavior of titanium-based alloys depending on their chemical compositions and immersion times.*

Keywords *Ti-Nb-Mo alloys, Ringer solution, corrosion*

1. Introduction

Titanium and its alloys are intensively studied in dental medicine due to their excellent biocompatibility, corrosion resistance and relatively low density [1]. Attempts to use titanium in the manufacture of implants date from the 1930 [2].

Metals such as Zr, Mo, Ni, Nb, Ta are elements that may be present in titanium alloys, as they exhibit excellent biocompatibility and belong to the group of non-toxic metals in their interaction with living tissue. Also, these elements are the most suitable alloying elements that can be added to decrease the modulus of elasticity without compromising its mechanical resistance [3-4]. In the case of titanium alloys, different methods of improving the corrosion resistance have been studied [5]. As an alloying element, niobium is known to be particularly for corrosion resistance in the simulated body fluid, also presenting a particularly good biocompatibility [6-7]. The use of molybdenum is still controversial, but some studies have shown that some Mo-containing Ti alloys can have adequate mechanical compatibility and good cytotoxic compatibility such as Ti-Mo, Ti-Mo-Ta, Ti-Mo-Zr-Fe [8-13]. The advantage of Mo is that it is a strong stabilizer of the β phase for Ti alloys in comparison to Nb, Ta or Zr [14-15]. Titanium alloys have been studied in different physiological mediums, such as artificial saliva, Hank solution, Ringer solution, NaCl solution to understand their electrochemical behavior as biomaterials subjected to corrosion in human organism [16-20]. In some studies, the influence of molybdenum content on corrosion resistance of Ti-Mo alloys in different simulated physiological environments has been investigated and one showed that the electrochemical behavior was improved due to the presence of molybdenum [21-26]. The superior properties of Ti-Nb and Ti-Nb-Zr systems are ascribed to addition of niobium (Nb) as the primary alloying element since it possesses distinguish attributes of improved β stability, low elastic modulus and high corrosion resistance [27-36].

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A recent concern of researchers is to find ways to optimize the properties of these alloys with applications in medicine. Thus, efforts are made to find an optimal surface properties of alloys that meets excellent qualities due to which the lifetime of the alloys can be improved in the aggressive environment of the human organism. Ti-based alloys remain some of the most widely used biomaterials. These studies described above were conducted to better understand the electrochemical behavior and their possible effects on the human environment.

The purpose of this study is to highlight the influence of different process parameters, such as pH of the electrochemical medium, chemical composition of alloys, immersion time on the corrosion behavior of investigated alloys.

2. Materials and methods

Samples from 5 alloys were used in this study, symbolized as follows: Ti12Mo, Ti10Mo8Nb, Ti8Mo16Nb, Ti6Mo24Nb and Ti4Mo32Nb. These alloys were synthesized in the specialized laboratory of the ISCR/C-Met-Site INSA Rennes (France) using the induction levitation-melting processing [37].

From as-cast Ti alloys mini-ingots, samples for electrochemical tests having an active area of 0,28 cm² were prepared by machining, polishing and mirror-like polishing. Before each test, the electrochemical surface of the alloys was sanded with SiC 1000, 2000, and 4000 grains of paper, then rinsed with distilled water and placed in the ultrasonic bath.

The electrochemical tests were performed at room temperature using the Ringer solution (1L solution of Ringer solution: Sodium chloride 8.60 g, Potassium chloride 0.30 g, Calcium chloride dihydrate 0.33 g.) at the natural pH ($pH = 6.5$), then Ringer solution at basic pH ($pH=8$) and acidic pH ($pH=4$). HCl and NaOH were used to modify the pH of initial Ringer solution.

To conduct the electrochemical tests, a glass electrochemical cell was used, with a simple wall and a potentiostatic assembly consisting of a working electrode, a reference electrode (SCE) and a platinum auxiliary electrode. The potentiostat used was PGZ-301. The experimental data was processed with the Voltmaster 4 software. EIS data processing (electrochemical impedance spectroscopy) was performed using the Zsimview program.

The tests were performed by scanning the potential with scan rate of 10mV/s, the applied potential being between -500÷2000mV. Tafel analysis was applied to extract the E_{corr} corrosion potential and the corrosion current density j_{corr} .

EIS spectra were recorded for the frequency range between 100 kHz and 25mHz, with a potential amplitude of 10mV and 20 points per decade.

3. Results and discussions

Titanium and its alloys are passive metallic materials in aqueous media. On the surface of bodies from titanium-based metallic materials is spontaneously formed an oxide film characterized by compactness, adherence at substrate and thinness. In contact with aqueous media the features of oxide film changes because thermodynamic and kinetic conditions are changed. The corrosion behavior of titanium-based metallic materials in aqueous electrolytes, and therefore also in natural or simulated biological environments, is depending to passive state for the given conditions.

Using electrochemical methods, the corrosion behavior of some alloys from the Ti-Mo-Nb system in Ringer solution was investigated, thus the use potential of these alloys as biomaterials being preliminary evaluated. The results of the electrochemical tests highlighted the relationship between corrosion parameters (E_{OCP} , E_{corr} , i_{corr} , R_p , R) and process parameters (Nb/Mo ratio, pH and immersion time (IT)).

3.1. Open circuit potential monitoring

Although by the monitoring the values of open circuit potential (E_{OCP}) in time the information about corrosion behavior is minimal, the passivity state can be qualitatively evaluated in the case of

metallic materials. In Figure 1(a,b,c) are shown the E_{OCP} values vs. immersion time (IT), and in Table 1 the E_{OCP} values at the end of monitoring time.

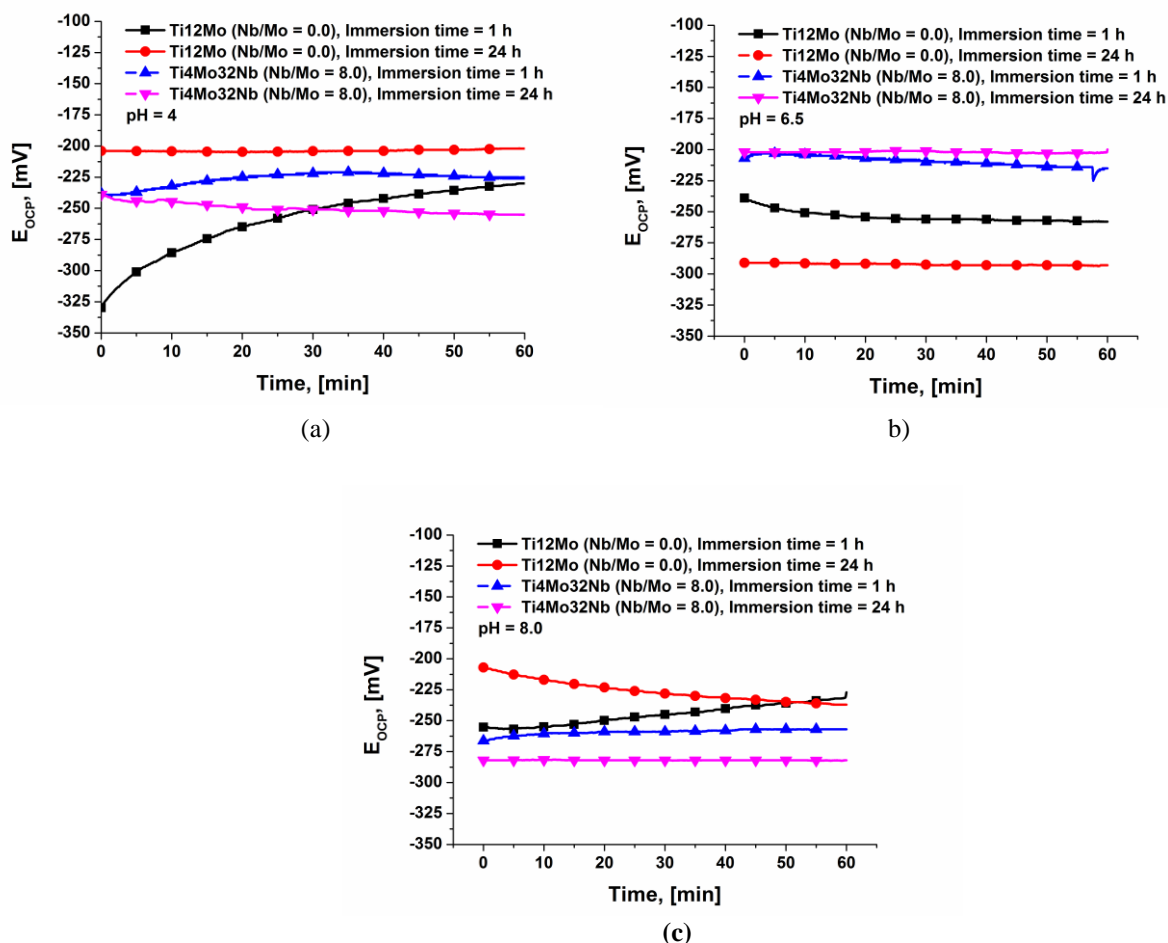


Figure 1. The evolution in time of the E_{OCP} values in Ringer solutions having different pH values: a) pH = 4.0; b) pH = 6.5; c) pH = 8.0.

Table 1. The E_{OCP} values of the end of immersion times

Alloys (Nb/Mo)	pH					
	4.0		6.5		8.0	
	IT, [h]					
	1 h	24 h	1 h	24 h	1 h	24 h
E_{OCP} , [mV]						
Ti12Mo (0.0)	-230.0	-202.0	-258.0	-293.0	-227.0	-237.0
Ti10Mo8Nb (0.8)	-237.7	-226.0	-213.9	-227.0	-221.0	-200.3
Ti8Mo16Nb (2.0)	-233.0	-270.0	-225.0	-237.0	-258.0	-264.0
Ti6Mo24Nb (4.0)	-211.0	-228.2	-251.0	-236.7	-214.0	-260.0
Ti4Mo32Nb (8.0)	-255.0	-255.0	-215.0	-199.8	-257.0	-262.0

From Figure 1(a,b,c) one can observe that the E_{OCP} values are quasi-stable in time even after 24 h of immersion, with some exception for initial immersion. This evolution indicates that passive films formed on the surface of all Ti alloys, regardless pH values and immersion times are stable.

3.2. Analysis of the corrosion behavior using the potentiodynamic polarization

Potentiodynamic polarization tests allows to estimate the values of some corrosion parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}) and for corrosion characterized by passivity, breakdown potential (E_{bd}), passive current density (i_{pas}). Figure 2(a,b,c) shows the characteristic potentiodynamic polarization curves and Table 2 and Table 3 contains the values of

corrosion parameters E_{corr} and i_{corr} . The polarization curves have a typical form for passive metallic materials.

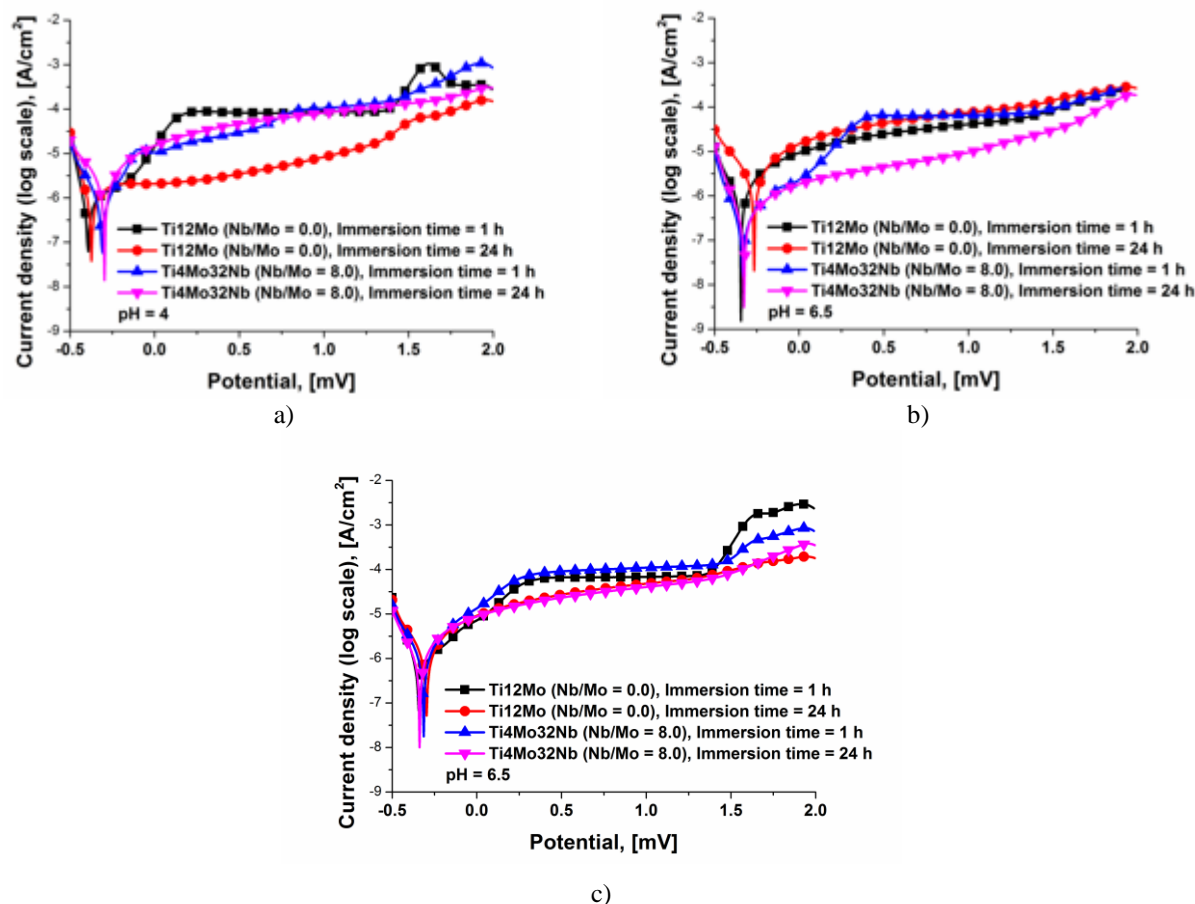


Figure 2. The polarization curves in Ringer solutions having different pH values: a) $pH = 4.0$; b) $pH = 6.5$; c) $pH = 8.0$.

The shape of polarization curves is characteristic for the titanium-based alloys immersed in simulated body fluids. The curves contain an active-passive transition followed through a passive domain of potential. It is worthy to mention that, after a 24 h immersion, regardless to alloy and pH , the passivation current density has a slight tendency to increase with the potential. This fact indicates that in time the passive films supports changes concerning structure and thickness. For short immersion time (1 h), especially for $pH = 8$, a well-defined potential plateau for which passivation current density has a value almost constant can be observed. It can be supposed that the passive films, as immersion time increase, change its features linked to corrosion behavior. Also, for $pH = 8.0$ the passivation current densities have values much closer. The values of E_{corr} are less than the values of E_{OCP} that is possible due to potentiodynamic polarization. The corrosion current density for $pH = 4$ and $pH = 6.5$ increased when immersion time has increased, and, inversely, for $pH = 8$ the values of this corrosion parameter has decreased.

Table 2. The measured values of E_{corr} versus experimental parameters used for the potentiodynamic polarization testing

Alloys (Nb/Mo)	pH					
	4.0		6.5		8.0	
	Immersion time, [h]					
	1 h	24 h	1 h	24 h	1 h	24 h
E_{corr} , [mV]						
Ti12Mo (0.0)	-387.7	-366.3	-337.3	-335.1	-334.0	-355.5
Ti10Mo8Nb (0.8)	-262.6	-255.4	-277.0	-256.9	-269.2	-251.2
Ti8Mo16Nb (2.0)	-375.4	-360.1	-408.1	-349.9	-315.3	-349.9
Ti6Mo24Nb (4.0)	-197.7	-151.2	-367.7	-383.6	-302.0	-383.6
Ti4Mo32Nb (8.0)	-301.7	-286.2	-326.9	-318.4	-305.6	-318.4

Table 3. The measured values of i_{corr} versus experimental parameters used for the potentiodynamic polarization testing

Alloys (Nb/Mo)	pH					
	4.0		6.5		8.0	
	Immersion time, [h]					
	1 h	24 h	1 h	24 h	1 h	24 h
i_{corr} , [$\mu\text{A}/\text{cm}^2$]						
Ti12Mo (0.0)	0.1622	0.5997	0.4239	0.8765	0.6511	0.4482
Ti10Mo8Nb (0.8)	0.0980	0.5335	0.3394	0.5316	0.2843	0.1698
Ti8Mo16Nb (2.0)	0.0586	0.3062	0.1920	0.2895	0.5244	0.2895
Ti6Mo24Nb (4.0)	0.0826	0.0679	0.1280	0.2670	0.3833	0.1667
Ti4Mo32Nb (8.0)	0.9124	1.3221	0.1977	0.2566	1.1044	0.2566

3.3. Analysis of corrosion behavior using electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy was performed under the potential conditions in the open circuit. The results were represented as the Bode diagrams, both *Impedance module vs. Frequency* and *Phase angle vs. Frequency*. Some Bode diagrams are shown in the Figure 3, and values of electrical quantities from equivalent electric circuit (EEC) are given in Table 4, Table 5, and Table 6. All EIS spectra were fitted using an R(QR) EEC.

EIS method used in corrosion bring out the structure and properties of passive films in relation with its protective features against corrosion of the substrate. For all investigated alloys, regardless the experimental condition of EIS tests, the spectra were fitted using an equivalent electric circuit identifies by R(QR). This EEC is characterized by a single time constant, which shows that passive film has a monolayer structure. Having in view the EEC (R(QR)), from its electric parameters, the thickness of passive films can be deduced using the following equations [38, 39]:

$$C_{\text{eff}} = ((Q \cdot R)^{\frac{1}{n}}) / R \quad (1)$$

$$d = \frac{\varepsilon \cdot \varepsilon_0}{C_{\text{eff}}} \quad (2)$$

Using data from Tables 4, 5 and 6, $\varepsilon_0 = 8.8542 \cdot 10^{-14}$, [F/cm], and e from literature, the thickness d , [nm] was evaluated for each EIS tests (Tables 4, 5 and 6).

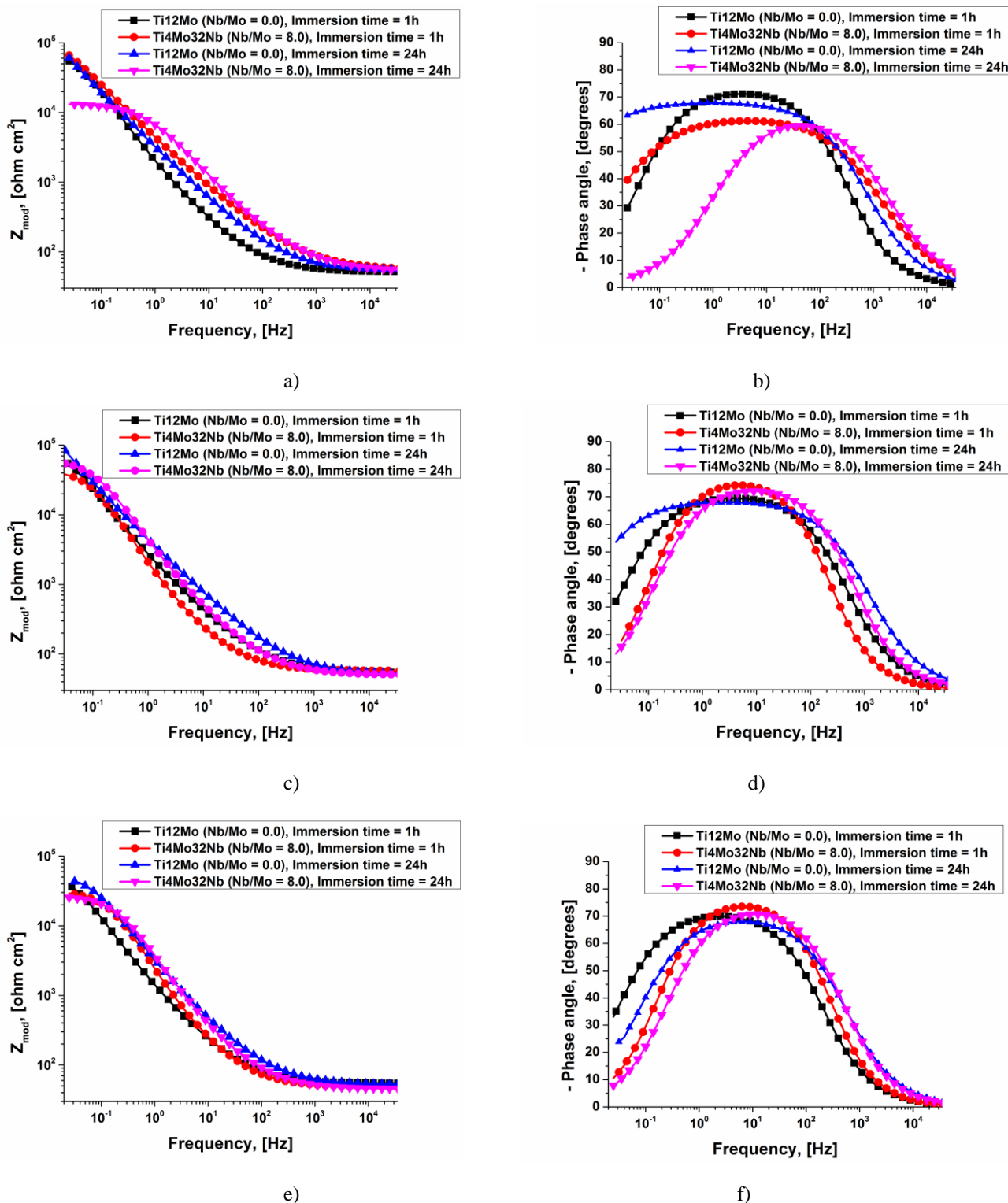


Figure 3. Bode plot of EIS spectra: Z_{mod} vs. Frequency: a) $pH = 4.0$, c) $pH = 6.5$, e) $pH = 8.0$;
 -Phase angle vs. Frequency: b) $pH = 4.0$, d) $pH = 6.5$, f) $pH = 8.0$

From shape of Bode plots and data inside within the tables, a slight difference between electrochemical behavior of investigated alloys can be observed. With the increase of pH , the Bode plots have shapes much more like, and the thickness of the passive films are more uniform. At acidic values of pH in relation with the chemical composition of alloys and immersion time, the shape of *Phase angle vs. Frequency* plots are more different. In other words, the more acidic pH , the more

evident are differences in electrochemical behavior in relation with chemical composition and immersion time.

Table 4. Values of the electrical parameters of EEC for investigated Ti-based alloys, pH = 4.0

Alloys (Nb/Mo)	R_s , [W·cm ²]	Q , [mS·sec ⁿ /cm ²]/ d, [nm]	n	R, [kW·cm ²]	$c^2/10^{-2}$	EEC
after 1 hour of immersion						
Ti12Mo (0.0)	54.10	11.64 / 6	0.77	88.340	0.96	R(QR)
Ti10Mo8Nb (0.8)	47.43	52.97 / 1	0.85	86.640	0.40	R(QR)
Ti8Mo16Nb (2.0)	57.02	37.55 / 2	0.85	72.300	0.41	R(QR)
Ti6Mo24Nb (4.0)	46.56	12.92 / 6	0.75	73.100	0.69	R(QR)
Ti4Mo32Nb (8.0)	55.39	31.44 / 2	0.87	31.380	0.63	R(QR)
after 24 hours of immersion						
Ti12Mo (0.0)	54.23	10.63 / 4	0.76	493.300	0.29	R(QR)
Ti10Mo8Nb (0.8)	49.43	39.35 / 1	0.79	60.630	0.37	R(QR)
Ti8Mo16Nb (2.0)	45.95	29.72 / 2	0.82	31.950	0.63	R(QR)
Ti6Mo24Nb (4.0)	50.43	25.60 / 2	0.78	82.450	0.43	R(QR)
Ti4Mo32Nb (8.0)	52.00	26.00 / 3	0.84	26.770	0.68	R(QR)

Table 5. Values of the electrical parameters of EEC for investigated Ti-based alloys, pH = 6.5

Alloys (Nb/Mo)	R_s , [W·cm ²]	Q , [mS·sec ⁿ /cm ²]/ d, [nm]	n	R, [kW·cm ²]	$c^2/10^{-2}$	EEC
after 1 hour of immersion						
Ti12Mo (0.0)	52.75	12.90 / 6	0.79	29.420	0.31	R(QR)
Ti10Mo8Nb (0.8)	53.81	34.19 / 2	0.88	89.220	0.36	R(QR)
Ti8Mo16Nb (2.0)	54.84	29.63 / 2	0.84	56.300	0.39	R(QR)
Ti6Mo24Nb (4.0)	54.75	22.24 / 3	0.76	57.800	0.67	R(QR)
Ti4Mo32Nb (8.0)	57.43	31.91 / 2	0.87	45.150	0.50	R(QR)
after 24 hours of immersion						
Ti12Mo (0.0)	53.10	12.00 / 7	0.75	46.400	0.59	R(QR)
Ti10Mo8Nb (0.8)	45.60	29.40 / 2	0.80	41.890	0.45	R(QR)
Ti8Mo16Nb (2.0)	59.73	19.54 / 3	0.82	86.000	0.61	R(QR)
Ti6Mo24Nb (4.0)	52.87	16.54 / 5	0.75	52.310	0.43	R(QR)
Ti4Mo32Nb (8.0)	50.30	19.02 / 4	0.84	63.270	0.55	R(QR)

Table 4. Values of the electrical parameters of EEC for investigated Ti-based alloys, pH = 8.0

Alloys (Nb/Mo)	R_s , [W·cm ²]	Q , [mS·sec ⁿ /cm ²]/ d, [nm]	n	R, [kW·cm ²]	$c^2/10^{-2}$	EEC
after 1 hour of immersion						
Ti12Mo (0.0)	54.10	36.98 / 1	0.82	86.100	0.96	R(QR)
Ti10Mo8Nb (0.8)	47.43	24.08 / 2	0.87	944.800	0.39	R(QR)
Ti8Mo16Nb (2.0)	57.02	17.90 / 2	0.77	577.200	0.41	R(QR)
Ti6Mo24Nb (4.0)	46.56	30.10 / 1	0.84	387.100	0.69	R(QR)
Ti4Mo32Nb (8.0)	55.39	30.40 / 1	0.75	925.000	0.63	R(QR)
after 24 hours of immersion						
Ti12Mo (0.0)	54.23	29.00 / 2	0.76	140.200	0.29	R(QR)
Ti10Mo8Nb (0.8)	49.43	28.69 / 2	0.81	53.500	0.37	R(QR)
Ti8Mo16Nb (2.0)	45.95	16.40 / 3	0.75	250.900	0.63	R(QR)
Ti6Mo24Nb (4.0)	50.43	27.90 / 2	0.82	91.200	0.43	R(QR)
Ti4Mo32Nb (8.0)	52.00	19.87 / 3	0.75	134.800	0.68	R(QR)



4. Conclusions

As a result of this research on the corrosion behavior of five alloys from Ti-Mo-Nb system in Ringer solution with various values of pH and for the different immersion times can be formulated the following conclusions.

The more acidic values of pH , the more evident are differences between corrosion behavior of titanium-based alloys depending on their chemical compositions and immersion times. For pH of 4.0 and a short immersion time the corrosion current densities were the lower. Increasing immersion time, the values of this corrosion parameter have increased. Inversely, at basic value of pH , the corrosion current densities have slightly decreased when immersion time increased.

Regardless of pH and immersion times, both lower corrosion current densities and lower thicknesses of passive films were found for alloys which have Nb/Mo values within 0.8 and 4.0.

Acknowledgements

The authors thank Prof. Thierry Gloriant for the support provided in the synthesis of titanium-based materials at ISCR/C-Met-Site INSA Rennes (France).

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Manuscript received: 27.05.2020