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## Corrosion hampering measurements of AISI 316 SS submerged in alkaline solutions

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### Abstract

The aim of this study was to investigate the corrosion behaviour of AISI 316 stainless steel to be used as rebars in construction. Austenitic stainless steel AISI 316 samples were put to the alkaline aqueous environments of composition: 0.01M NaOH+0.01M KOH both free of chlorides and those containing 3% NaCl. The hydrogen ion exponent pH was 10 and 12.5. The following electrochemical methods have been used to investigate corrosion: polarization curves PC, Electrochemical Impedance Spectroscopy EIS, and 24-hour open circuit potential OCP studies. The obtained results are quite comprehensive and indicate on faster self-restoration of passive films under action of chlorides. The chlorides do not cause essential changes in the potential values, both corrosion and critical, in comparison with the results on samples studied in the solutions free of chlorides. The studies reveal that, out of two alkaline environments, the AISI 316 SS better performs in the presence of solutions of lower pH. The presence of chlorides in the solution enables faster formation of a passive film and its eventual auto-restoration in case of disruption. The presented investigation results indicate the chlorides accelerate transition of Cr(III) to Cr(VI) on steel surface. The longer steel AISI 316 sample remains in the solution, the more and more film formed on the steel surface strengthens and gets stabilized resulting in better surface protection.

**Keywords:** Corrosion, EIS measurements, AISI 316 SS, alkaline environments, chlorides.

### Pomiary korozji stali AISI 316 w roztworach alkalicznych

#### Streszczenie

W pracy przedstawiono wyniki badań korozyjnego zachowania się stali kwasoodpornej AISI 316 – przewidywanej do zastosowań konstrukcyjnych w budownictwie – w roztworach alkalicznych. Przygotowane próbki stalowe umieszczono w roztworach o składzie: 0.01M NaOH+0.01M KOH bez chlorków oraz zawierającym 3% NaCl. Wykładnik jonów wodorowych wynosił 10 i 12,5. Do pomiarów korozyjnego zachowania się stali zastosowano badania krzywych polaryzacyjnych PC, elektrochemiczną spektroskopię impedancyjną EIS, oraz badania potencjału obrotu otwartego OCP. Wyniki badań są zgodne i wskazują na szybsze samoodnowienie warstewki pasywnej pod wpływem chlorków. Chlorki nie powodują zasadniczych zmian w wartościach potencjału, i to zarówno korozyjnego jak i krytycznego, w porównaniu z wynikami uzyskanymi w roztworach bez chlorków. Wyniki badań wskazują, że spośród dwu zastosowanych roztworów, stal AISI 316 jest bardziej odporna na korozję w roztworach o niższym pH. Obecność chlorków w roztworze umożliwia szybsze tworzenie się warstewki pasywnej i jej odtwarzanie w przypadku przerwania. Wyniki badań wskazują, że chlorki przyspieszają przejście Cr(III) do Cr(VI) na powierzchni stali. Wydłużenie czasu pozostawiania stali w roztworze powoduje umocnienie warstewki i stabilizację ochrony powierzchni stali.

**Słowa kluczowe:** korozja, stal AISI 316, pomiar EIS, środowisko alkaliczne, chlorki.

### 1. Introduction

Type UNS S31600 stainless steel is the most popular acid-resistant material of increased resistance to corrosion due to Mo addition. It belongs to austenitic stainless steels and has a face-centered (fcc) structure [1, 2]. This steel is essentially nonmagnetic in the annealed condition, and possesses excellent cryogenic properties and good high-temperature strength. In comparison with AISI 304 SS in this steel molybdenum is added to increase halide pitting resistance. It may be used for machine parts working in temperatures up to 300-400 °C. Its frequent application has been found in pharmaceutical, textile, and food industries, as well as in cellulose and paint manufacture.

Corrosion resistance is often the most important characteristics of this type of stainless steels, and as the addition of Mo has been reported to improve this feature, the AISI 316 SS is used in chemical and marine industries, as well as in other environments containing chlorides. Recently it has been used in construction in the form of reinforcement bars (rebars) [3-5].

The aim of the paper was to investigate, measure and reveal the corrosion behaviour of AISI 316 stainless steel in alkaline environments without NaCl and with the addition of 3% NaCl. The consequences of this long-term stay and the behaviour of AISI 316 SS in this modified environment have been also investigated.

### 2. Experimental and Method

Nominal composition of AISI 316 stainless steel studied is given in Table 1.

Tab. 1. Chemical composition of AISI 316 SS [1]

Tab. 1. Skład chemiczny stali AISI 316 SS [1]

Element contents, wt%								
Cr	Ni	C	Mn	Si	P	S	Other	Fe
16-18	10-14	0.08	2	1	0.045	0.03	Mo 2-3	bal.

Most of the studies have been carried out at the Division of Electrochemistry in the *Instituto Superior Tecnico* Lisbon, Portugal. They were part of a bigger project.

#### 2.1. Preparation of samples

Square samples of the surface area of 1 cm<sup>2</sup>, cut out of a steel sheet 2 mm thick have been used to the studies. Each sample for corrosion studies was connected with a copper wire cable (Fig. 1). After removal of the isolation the wire ends were connected with the steel sample. A special high purity silver paint was used for this purpose. Two layers were put to ensure better conductivity of electric current.

After the silver paint was dry, EpoFix Kit was used to flood the samples. EpoFix Resin and EpoFix Hardener were mixed in ratio 15:2 parts of volume. Then all the probes were put into the cylindrical shape forms and flooded with the mixture. Next they were left for 24 hours to settle and dry. Before that, the forms were cleaned and lubricated with petroleum jelly just to make samples easier to take out when they are dry.

Hard dried samples were taken off the forms and then subjected to mechanical polishing. The steel samples were polished with waterproof silicon carbide paper with four of the grit sizes 320, 800, 1200, and 4000.

Afterwards the samples were put to the buffing process using polishing wheel/disc with diamond polishing compound of the graininess 3  $\mu\text{m}$ , and  $\frac{1}{4}$   $\mu\text{m}$ , respectively.

In view of avoidance of pitting corrosion, a two-component tough adhesive Araldid Rapid of Ceys® was used. After this operation the bee wax was applied on the sample edges to protect them against corrosion. Thus the samples active area was diminished by 1 mm at each side, leaving the open area of 8×8 mm.

## 2.2. Preparation of solutions

The comparative corrosion studies of such prepared the AISI 316 stainless steel samples have been carried out in four alkaline solutions prepared on de-ionized water. They were mixed as follows:



and it was the first one to carry the corrosion experiments. The second solution was different by introducing chlorides to the composition (1), adding 3% sodium chlorides:



Additionally these two solutions (1) and (2) were differing with two pH values in each of them. Lower pH was established at 10, whereas the higher one was more alkaline and equaled 12.5.

Proper pH of the solutions were corrected by addition of different substances: NaOH to increase the alkalinity from 12 to 12.5, and nitric acid  $HNO_3$  to lower pH down to 10.

## 2.3. Set-up for corrosion studies

Scheme of the measurement cell is presented in Fig. 1. The electrochemical cell consisted of a working electrode WE (steel sample), and counter electrode as the platinum reference electrode with submerged electrolytic key/bridge, which formed soluble connection between the cell and saturated calomel reference electrode (SCE). The aim of the electrolytic key was to avoid getting chlorides from the reference electrode to the solution in view of getting correct experimental results.

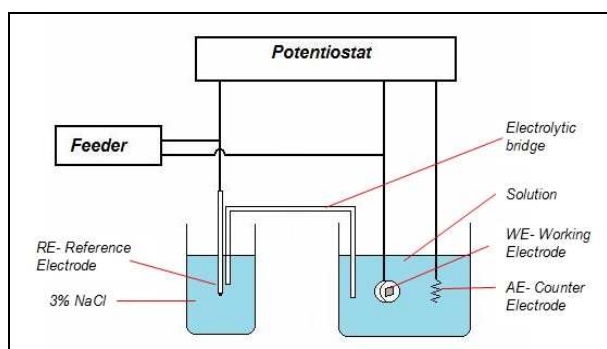


Fig. 1. Electrochemical cell schematic  
Rys. 1. Schemat stanowiska badawczego

The studies of corrosion behaviour of AISI 316 SS were carried out in alkaline aqueous solutions. Open circuit potential (OCP) was measured by means of potentiostat VoltaLab PGZ100 and computer with Windows 2000 and the software VoltaMaster 4 used to serve the potentiostat. The electrochemical cell was placed inside the Faraday cage.

Each OCP experiment lasted for 24 hours. The potentiostat was programmed to read out the potential value every 2 seconds. At that reading frequency, 64000 points were recorded, and it was maximum value, limited by the software. The measurement started at once after putting the sample to the solution [6].

Polarization curves and electrochemical impedance spectroscopy (EIS) were obtained with the use of Gamry Instruments with Reference 600-Potentiostat/Galvanostat/ZRA, connected with computer and operational system Windows 2000 and the software.

Gamry Instruments Framework, version 4.35 (2005) was used to obtain polarization curves. Before starting measurements the samples were left in the solution for 30-minute stabilization, during which the open circuit potential (OCP) was measured.

The electrochemical impedance spectroscopy (EIS) results were also obtained with the use of Gamry Instruments Framework. The samples were left in the solution for one hour before starting with the first test. The following measurements were carried out after the time of: 3 hours, 6 hours, 12 hours, 24 hours, 48 hours, and 72 hours. Before each of the tests the instruments were set up for 5-minute potential measurement.

## 3. Results of the studies

### 3.1. Open circuit potential (OCP)

In Fig. 2 the open circuit potential (OCP) results are presented.

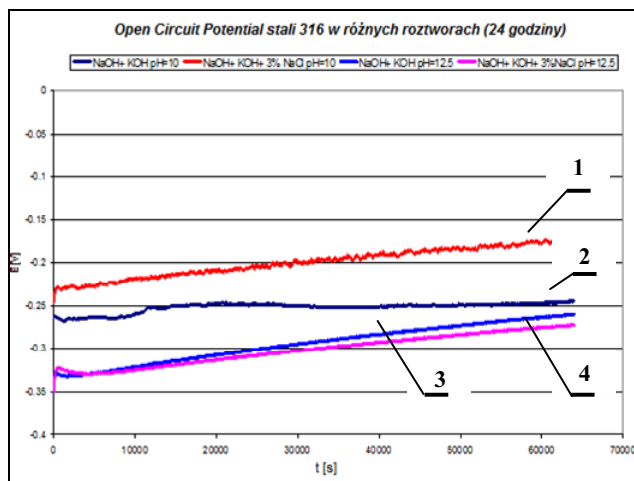


Fig. 2. Open circuit potential received during 24 h on AISI 316 SS sample in different solutions: 1 – NaOH+KOH, pH=10; 2 – NaOH+KOH+ 3%NaCl, pH=10; 3 – NaOH+KOH, pH=12.5; 4 – NaOH+KOH+ 3%NaCl, pH=12.5  
Rys. 2. Potencjały obwodu otwartego OCP otrzymane po 24 h na próbkach stali 316 SS w różnych roztworach: 1 – NaOH+KOH, pH=10; 2 – NaOH+KOH+ 3%NaCl, pH=10; 3 – NaOH+KOH, pH=12.5; 4 – NaOH+KOH+ 3%NaCl, pH=12.5

As can be seen (Fig. 2), the open circuit potential (OCP) of the electrode studied at higher pH = 12.5 (curves 3, 4), both with NaCl (4) and without it (3), has been increasing steadily and approaching the value of OCP potential of the sample submerged in the solution of pH = 10 without chlorides (2). Some hesitation, deterioration and higher deflection of the curve 1 in Fig. 2, for sample in the solution of pH = 10, containing 3% NaCl, could be caused by an inaccuracy in sample connection. On the other hand, the AISI 316 SS in alkaline solution of pH = 10 after a short time has been almost flat and stable for a long time, and at the end of the study started to increase slightly above  $-0.25$  V vs. SCE.

### 3.2. Polarization curves (PC)

In Fig. 3 the corrosion results of polarization curves (PC) of the AISI 316 SS in different alkaline solutions have been presented. One may notice two groups of PC curves of different corrosion potentials. The series presenting behaviour of samples submerged in the solutions of pH = 10, both without chlorides and with the addition of 3% NaCl (curves 1, 2 in Fig. 3), indicate the same corrosion potential  $E_{cor}$  shifted of about 0.2 V in positive direction, above the potential of samples left in the solutions of pH = 12.5 (curves 3, 4 in Fig. 3).

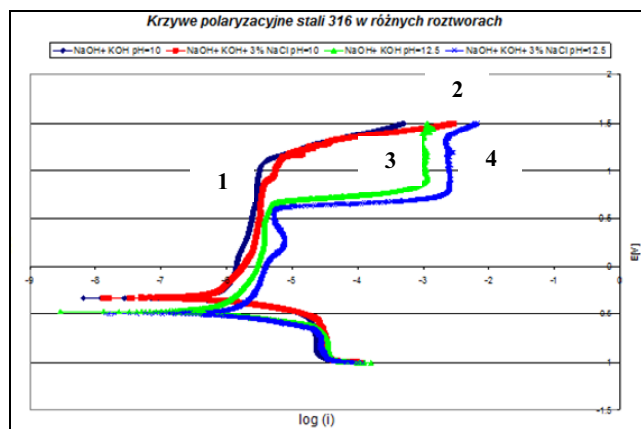


Fig. 3. Polarization curves obtained on AISI 316 SS samples in different solutions: 1 – NaOH+KOH, pH=10; 2 – NaOH+KOH+3%NaCl, pH=10; 3 – NaOH+KOH, pH=12.5; 4 – NaOH+KOH+3%NaCl, pH=12.5

Rys. 3. Krzywe polaryzacyjne PC otrzymane na próbkach stali 316 SS w różnych roztworach: 1 – NaOH+KOH, pH=10; 2 – NaOH+KOH+ 3%NaCl, pH=10; 3 – NaOH+KOH, pH=12.5; 4 – NaOH+KOH+ 3%NaCl, pH=12.5

Some changes in the passive state may be observed on AISI 316 SS submerged in the solution containing NaCl of pH=12.5 (the potential range of 0 V to 0.4 V on curve 4) and it is probably the electro-oxidation of chromium from Cr(III) to Cr(VI). That behaviour may indicate the protective oxide film on the steel surface has been losing its tightness, but after crossing the potential 0.7 V vs. SCE it gets self-restoration by re-gaining protective properties. All this is due to the presence of chlorides which facilitate and accelerate the oxide film formation on the steel surface.

### 3.3. Electrochemical Impedance Spectroscopy (EIS)

The impedance Bode plots (Figs. 4-7) have been performed to better understand the processes occurring on AISI 316 SS samples inside the electrochemical cell filled up with consecutive varying aqueous alkaline solutions.

The first conclusion coming out of the Bode plots (Figs. 4-7) on the corrosion behaviour of AISI 316 SS is that the material remaining longer in the solution possesses more stable oxide film. The change of phase angle  $\theta$  in the logarithmic function of angular frequency  $\omega$  (curve B) well reflects the dependence. The curves of samples remaining in the solution for over 24 hours, after achieving maximum value of phase angle  $\theta$  decrease much slighter than the function of the sample submerged in the solution for one hour.

Lines B of the studied samples (Figs. 4-7) after one hour of the submerge of samples in the solution when obtaining maximum value begin to fall down much sharper/stronger than the same lines left in the solution for a longer time. However, the corrosion does not proceed because the reactance (Lines A) is steadily growing do not achieving constant values that does not prove of the corrosion. The course of function  $\log|Z| = f(\log(\omega))$  proves of a high resistance of this sort of steel.

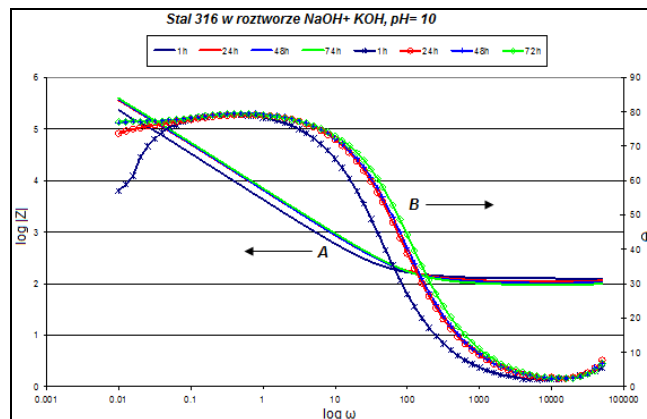


Fig. 4. Electrochemical impedance spectroscopy plots obtained on AISI 316 SS samples in the solution NaOH+KOH, of pH=10 (Bode plots); timing from 1 h to 72 h

Rys. 4. Wykresy impedancyjne EIS otrzymane na próbkach stali 316 SS w roztworze NaOH+KOH, pH=10 (wykresy Bode'go); czas doświadczenia 1 h do 72 h

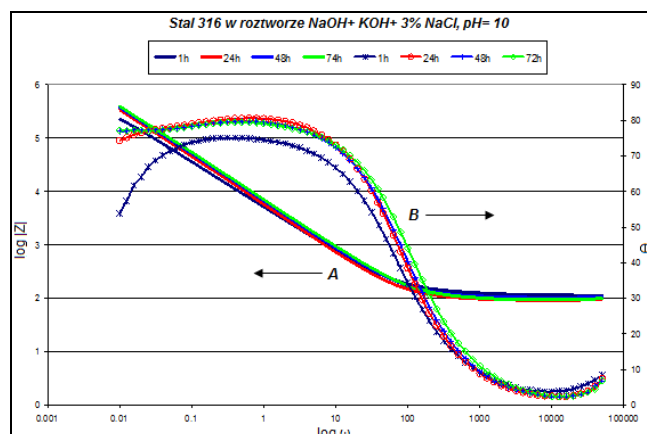


Fig. 5. Electrochemical impedance spectroscopy plots obtained on AISI 316 SS samples in the solution NaOH+KOH+3%NaCl, of pH=10 (Bode plots); timing from 1 h to 72 h

Rys. 5. Wykresy impedancyjne EIS otrzymane na próbkach stali 316 SS w roztworze NaOH+KOH+3%NaCl, pH=10 (wykresy Bode'go); czas doświadczenia od 1 h do 72 h

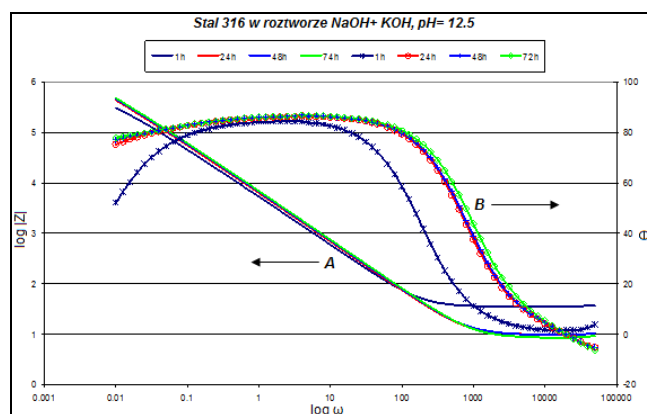


Fig. 6. Electrochemical impedance spectroscopy plots obtained on AISI 316 SS samples in the solution NaOH+KOH, of pH=12.5 (Bode plots); timing from 1 h to 72 h

Rys. 6. Wykresy impedancyjne EIS otrzymane na próbkach stali 316 SS w roztworze NaOH+KOH, pH=12.5 (wykresy Bode'go); czas doświadczenia od 1 h do 72 h

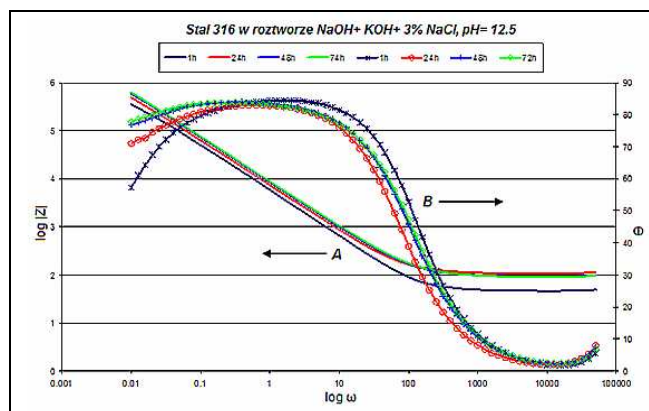


Fig. 7. Electrochemical impedance spectroscopy plots obtained on AISI 316 SS samples in the solution NaOH+KOH+3%NaCl, of pH=12.5 (Bode plots); timing from 1 h to 72 h

Rys. 7. Wykresy impedancyjne EIS otrzymane na próbkach stali 316 SS w roztworze NaOH+KOH+3%NaCl, pH=12.5 (wykresy Bode'go); czas doświadczenia od 1 h do 72 h

## 4. Discussion

In the initial phase of investigation the Luggin capillary served to prevent the direct contact of the reference electrode. Into the capillary the same solution was poured that was actually in the cell and the reference electrode was placed inside. This way the KCl solution of electrode was diluted against its getting to the solution in the cell which occurred through the capillary membrane. During performing of short tests, such as polarization curves, this method is very efficient because the KCl concentration inside the capillary is so low that it has no significant effect on result. The problem arises during long-term tests where there is no possibility for constant supervision over their performance, e.g. OCP which was measured throughout 24 hours without break. During night hours the electrolyte in the capillary was leaking to the solution resulting in self-breaking the test self-acting due to flow connection of the electrolyte with the cell. That fact decided to replace the Luggin capillary into the electrolytic key [7, 8].

Electrochemical Impedance Spectroscopy shows the reaction of the system on sinusoidal interference of electrode potential. The oxide film which is formed on the AISI 316 SS surface under action of the solution is very resistant with small number of pores resulting in very high resistance value [9, 10].

Observing presented Bode plots (Fig. 5 and Fig. 7) one may notice that for steel samples placed in electrolytes containing 3% NaCl the capacitance begins to grow for higher values of frequencies and lower impedance values. For steel samples submerged in solutions free of chlorides (Fig. 4, and Fig. 6) the resistance begins to grow for the frequency values of about 100 Hz and impedance of about 2  $\Omega$ , whereas in Fig. 7, under action of solution containing 3% NaCl the frequency for which the resistance begins to grow equals about 1000 Hz, and impedance of about 1  $\Omega$ . Such a situation happens due to chlorides which accelerate steel oxidation and with this a passive protective film is formed resulting in the increase of corrosion resistance of steel [8, 11].

Looking onto the function dependence of phase angle  $\theta$  on the angular frequency  $\omega$  one may notice the function falls in the last stadium. For samples left in electrolytes just for an hour that fall is much greater than that measured after 24 hours. That means the longer sample is left in the solution, the film formed on the surface is tougher and gets stabilized resulting in better surface protection. The falls reach similar values of phase angle. The steel surface is more stable for the phase angle  $\theta$  of about 80°, whereas for measurements after one hour one may notice the function decreases more sharply for the sample dipped/plunged in the solution of higher pH = 12.5 (Fig. 6, and Fig. 7) achieving lower final values of phase angle (of about 60°) than the sample in solutions of pH = 10.

One may compare the electrochemical research results presented by the authors of reference [12] with the measurement results obtained in this work on the AISI 316 SS samples and find they are quite comprehensive. The similarity of the obtained results proves of a correct approach to the studies presented in this work.

## 5. Conclusions

In the corrosion studies of austenitic AISI 316 stainless steel the hampering effects of alkaline solutions were measured. Based on the study results, the following conclusions may be drawn:

- (1) The obtained corrosion study results are quite smooth, and even at high potentials no sharp changes are noticeable. The difference between the critical potential and the corrosion potential for AISI 316 SS equals some hundreds of millivolts in all cases; therefore pitting is practically impossible.
- (2) The contents of 3% of sodium chloride in the solutions do not affect changes in the values of potentials (corrosive nor critical) in comparison with the results of tests carried out in the solutions free of chlorides. On the other hand, the corrosion resistance is highly affected by pH of solution: better resistant to the solutions of lower pH.
- (3) The presence of chlorides is of benefit at prompter formation of passive film and its eventual auto-restoration in case of damage. The chlorides accelerate transition of Cr (III) to Cr (VI). This phenomenon is apparent in the form of wave on the polarization curves plots of steel subjected to the action of solution containing chlorine ions, especially in the solution of pH = 12.5 (range of 0 mV to 500 mV).
- (4) One may conclude from OCP and EIS plots that the protective oxide film has been stabilized in time lapse during the sample submerged in the solution. The open circuit potential OCP measurement results confirm that the austenitic stainless steels in alkaline environments behave passively.
- (5) Very similar conclusions to the OCP results may be drawn based on the impedance dependences (function of the phase angle  $\theta$  on the angular frequency  $\omega$ ). For samples left in the solutions for only one hour, the function falls in the last stadium to lower values than at measurements during 24 hours. That means the longer AISI 316 SS sample is left in the solution, the more film formed on the steel surface is enhanced and stabilized leading to better protection of the surface.
- (6) In measurements of impedance the obtained values of resistance exclude a possibility to arise pitting at the applied parameters of the process. On the Bode plots, presenting the ratio of frequency to resistance it is visible that the function grows steadily and does not stabilize (there is one constant in the plots). That means the properties of pore walls and bottom are equal/identical. The oxide/hydroxide film formed on the AISI 316 SS sample in alkaline environments is tight, compact, and well adheres to the substrate.

*The authors wish to thank Dr Fatima Montemor and Prof. Joao Fernandes from the Instituto Superior Tecnico Lisboa, Portugal, for their guidance and useful discussion during the experimental studies. We acknowledge also the availability of facilities of the Laboratory of Electrochemistry, IST Lisbon.*

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## INFORMACJE

# Targi Automatyki i Pomiarów Automaticon 2010

W dniach 23-26 marca 2010 r. odbyły się po raz szesnasty Targi Automaticon. Zostały one zorganizowane przez Przemysłowy Instytut Automatyki i Pomiarów PIAP oraz firmę MVM na terenie EXPO XXI w Warszawie. W targach wzięło udział w charakterze wystawców ponad 300 firm z branży automatyki, robotyki, mechatroniki, elektroniki i pomiarów. Większość z nich uczestniczyła także w poprzednich targach Automaticon, ale pojawiły się też firmy nowe.

W trakcie targów była możliwość zapoznania się z aktualną ofertą produkcyjną wielu firm branżowych, a także z nowymi produktami i technologiami obecnymi już na rynku światowym. Naszą uwagę zwróciły stoiska PIAP, EM test, Astat, Fanuc, Wenglor, Harmonic Drive, a zwłaszcza PBT Technik, prezentująca maszyny do montażu powierzchniowego: ręczne, półautomatyczne, automatyczne oraz do inspekcji optycznej płytek: mikroskopy i wizyjne systemy pomiarowe. Na targach były widoczne silne tendencje rozwoju robotów i automatyzacji procesów.

Równoległe z targami odbywały się seminaria naukowe dotyczące zagadnień związanych m.in. z systemami sterowania i przemysłowymi sieciami komunikacyjnymi, robotami przemysłowymi, kompatybilnością elektromagnetyczną i transmisją bezprzewodową. Jedno z nich zostało poświęcone projektowi Młodzi Innowacyjni, gdzie młodzi ludzie prezentowali swoje prace dyplomowe: inżynierskie, magisterskie i doktorskie.

XVI targi Automaticon stanowiły okazję do prezentacji nowoczesnych rozwiązań konstrukcyjnych, nawiązywania kontaktów między firmami i klientami, inspirowania wyobraźni studentów i uczniów szkół technicznych, którzy licznie odwiedzali stoiska targowe.

Większa niż w ubiegłym roku obecność polskich firm, zwłaszcza małych i średnich, napawa optymizmem. Ten wzrost nastąpił przy zmniejszeniu się liczby obecnych dużych firm i korporacji międzynarodowych. Jest to oznaką dobrego radzenia sobie przez polskich przedsiębiorców ze skutkami światowego kryzysu gospodarczego.

Pierwszego dnia targów przedstawiciele prasy i wystawcy zostali zaproszeni na konferencję prasową, na której dokonano podsumowania przygotowań do targów i scharakteryzowano ich zasięg, w różnych przekrojach. Określono targi jako wyjątkowe z dwóch powodów. Pierwszym z nich jest trudna sytuacja

w gospodarce światowej i europejskiej. W ubiegłym roku wiele firm borykało się z kryzysem gospodarczym, co nie pozostało bez wpływu na branżę automatyki przemysłowej. Targi dały okazję do oceny, jak firmy duże i mniejsze poradziły sobie z kryzysem. Drugim elementem podkreślanym przez Organizatorów było oddanie do użytku nowo wybudowanej hali wystawienniczej na terenie EXPO XXI, która dała nowe możliwości targom. Dzięki niej zwiedzający mają przestronniejsze ciągi komunikacyjne oraz nie zachodzi już potrzeba ograniczania powierzchni stoisk wystawców ani roztawiania stoisk w holu czy salach konferencyjnych.

Wydawnictwo PAK wzięło udział w targach nie tylko jako patron medialny, ale również jako wystawca. Stoisko PAK znajdowało się w największej hali nr 1. Zaprezentowaliśmy Wydawnictwo PAK, miesięcznik *Pomiary Automatyka Kontrola*, a także wydane przez nas pozycje książkowe. Największy popyt miała nasza najnowsza książka „Mikrokontroler PIC w zastosowaniach” I. P. Kurytnika.

W dniu 23 marca 2010 r. odbyła się gala wręczenia Złotych Medalii targów Automaticon. Komisja konkursowa w składzie mgr inż. Przemysław Zbichorski, prof. dr hab. inż. Jan Maciej Kościelny (przewodniczący, redaktor działu Automatyka PAK), prof. dr hab. inż. Krzysztof Sacha, doc. dr inż. Stanisław Kaczanowski oraz mgr inż. Irma Pęciak przyznała wyróżnienia firmom:

- Aplisens S.A., za elektropneumatyczny ustawnik pozycyjny APIS,
- Fanuc Robotics Polska, za robota FANUC M-1iA,
- Festo Sp. z o.o., za liniowy napęd elektryczny EGC,
- Lumel S.A., za analizator / rejestrator parametrów trójfazowej sieci energetycznej NDI,
- Multiprojekt, za aktuator servotube XHA 3804,
- RECTUS Polska Sp. z o.o., za napędy pneumatyczne, obrotowe Puretorg z systemem przenoszenia napędu łopatkowego.

Wydawnictwo PAK składa Organizatorom gratulacje za dobrą organizację targów i dołożenie starań o utrzymanie wysokiej pozycji Targów Automaticon wśród najważniejszych wydarzeń branżowych.

Opracowanie: Beata KRUPANEK