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BHABHA ATOMIC RESEARCH CENTRE

ELECTROCHEMICAL NOISE FROM CORRODING CARBON
STEEL AND ALUMINIUM

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Materials Science Division

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60 Abstract : Electrochemical noise measurements were conducted on carbon steel and aluminium in sodium chloride solutions. Noise parameters like standard deviation of potential and current, noise resistance, pitting index, noise power were studied for the purpose of measuring corrosion rate. These parameters compared well with the corrosion rate. Pitting index was not very reliable. Current noise was more close to the corrosion rates. General corrosion gave rise to white noise type of power spectrum while flicker noise type of spectrum was obtained from pitting attack. Sodium nitrite is shown to inhibit the corrosion of carbon steel. Aluminium corrodes in the early period of exposure and passivates during long exposure.

70 Keywords/Descriptors : CARBON STEELS; ALUMINIUM; FITTING CORROSION;
ELECTROCHEMICAL CORROSION; CORROSION RESISTANCE; SODIUM NITRATES;
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INTRODUCTION

Periodic oscillation in current and potential signals from corroding specimens, referred to as electrochemical current noise (ECN) and electrochemical potential noise (EPN) respectively and collectively known as electrochemical noise (EN) has gained considerable attention in recent times. It is a rich source of information⁽¹⁾ on the electrochemical processes going on on the surfaces. Iverson's⁽²⁾ study of potential noise on platinum coupled with other materials was the first use of noise measurement in corrosion process. He reported low potential fluctuation in Pt-Pt couple compared to other galvanic couples involving Pt. Since then this technique has been used for many metals / alloys in various environments like: Iron-steel^(3,4,5,6,7,8) copper alloys^(9,10) aluminium alloys^(11,12) etc.

In the field of nuclear and thermal power generation, the current interest is on the development of on-line corrosion monitoring / surveillance systems. The additional demand on such a system is due to involvement of high temperature, high pressure and long duration besides low levels of measurable parameters. Macdonald et al.^(13,14,15) have studied electrochemical noise in subcritical and supercritical aqueous system (250^o C - 500^o C). Westinghouse Hanford Co, is currently testing its prototype corrosion monitor for double / single shell, high level nuclear wastes⁽¹⁶⁾. EN is also being tested for underground storage tanks for defence nuclear wastes⁽¹⁷⁾ In combination with other non-destructive techniques, EN is utilized for ageing evaluation⁽¹⁸⁾ of duplex stainless steel components of primary circuit of pressurized water reactors. Attempts are at Ontario Hydro (Canada) to

use electrochemical noise for corrosion monitoring of service water systems ⁽¹⁹⁾. Microbiologically induced corrosion (MIC) has been studied with electrochemical noise measurement by Little et al ⁽²⁰⁾. Fahley et al ⁽²¹⁾ have attempted monitoring of surface condition of zirconium in acidified chloride solutions in presence of oxidizing ions.

EN has shown good promise as a tool for corrosion monitoring, passivity and coating integrity, warning device prior to leakage etc. The specimens are not put under influence of any external potential or current signals, and corrode in the natural manner. The present paper reports some experimental results on the noise measurements carried out on carbon steel and aluminium in sodium chloride solutions. Carbon steel was tested in 1M NaCl, 0.01 M NaNO₂ and their mixture to provide corrosive, passive and inhibited environments. Aluminium provides an example of passive system but is susceptible to pitting corrosion in presence of chloride ions. The emphasis is on measurement of electrochemical noise and its interpretation in terms of corrosion rate and type of corrosion processes.

EXPERIMENTAL

Rectangular specimens of carbon steel (ASTM A102, C 0.11, Mn 0.35, Si 0.04, P 0.04, S 0.04) and aluminium 1S (Cu 0.05, Si 0.3, Fe 0.4, Mn 0.05, Zn 0.1, B 20 ppm) were mounted in Metset 101 epoxy resin so as to expose identical areas (1 cm²). The experimental setup is shown schematically in figure 1. Three identical electrodes were used together in one set of experiments: two (central and one on one side) for measuring galvanic current with a zero resistance ammeter (ZRA) and two (central and one on the other side) for measuring potential with a high input impedance electrometer amplifier. The third specimen is used as a

pseudoreference electrode. All specimens were polished to 600 grit paper, degreased and rinsed in water before use. A saturated calomel electrode was also used to monitor the potential of the couple. The distance between the electrodes was kept fixed at 1 cm. Carbon steel was exposed in 1M NaCl and 0.01 M NaNO₂ + 1M NaCl solution for ten days. Aluminium was tested in tap water, 0.01 M, 0.1 M and 1.0 M NaCl solutions up to 100 hours. Signals from ZRA and differential amplifier were fed into a 16 channel ADC controlled by a PC. Current and potential signals were recorded with a sampling rate of 1 Hz. Total number of sampled data points for each channel was 1024. Data analysis was performed off line in time domain with statistical analysis and frequency domain using FFT. Corrosion was monitored separately also by conventional polarization methods.

RESULTS AND DISCUSSION

1. CARBON STEEL

Figure 2 shows the variation in the potential of carbon steel with time in 1M NaCl and 0.01 M NaNO₂ + 1 M NaCl solutions. It was found that while the absolute potential (figure 2 a) was quite different when measured with calomel and the pseudoreference (carbon steel) electrode, their fluctuation, i.e. the standard deviation, std(v) was of the same order (figure 2b). Potential decreased from initial -600 mv to -700 mv in the first twenty four hours and then remained constant at that up to 100 hours followed by slow oscillations. The fluctuation in the potential, std(v) also followed the same pattern. The std(v) ranged from 0.005 mv to 0.012 mv except in the initial period when it was in the range 0.03 - 0.05 mv. The electrode potential represents the overall process on the surface while the std (v) represents local processes on the surface. The

pseudoreference electrode appears to be more suitable for a long duration experiment or on-line use when the standard reference electrodes tend to deviate. Figure 2 also shows the potential of carbon steel in 0.01 M NaNO_2 + 1M NaCl solution. Carbon steel samples were initially immersed in 0.01 M NaNO_2 solution. NaCl was added to 0.01 M NaNO_2 solution after 48 hours of immersion to adjust the concentration to 0.01 M NaNO_2 +1 M NaCl. The potential of the carbon steel was -250 mv initially which became more anodic with time, reached -96 mv in 48 hours. Addition of NaCl decreased the potential to -500 mv but it remained higher than that in 1 M NaCl alone. Figure 3 shows the galvanic current and fluctuation in the galvanic current noise, $\text{std}(i)$ with time. In most cases the galvanic current was in the range 0.4 to 0.5 $\mu\text{A}/\text{cm}^2$. Galvanic current and the $\text{std}(i)$ were low in 0.01 M NaNO_2 solution and was increased by addition of NaCl but remained lower than that in 1M NaCl alone. Figure 4 shows the variation in the noise resistance, $R_n^{(22)}$ defined as the ratio of the $\text{std}(v)$ to $\text{std}(i)$, the polarization resistance, R_p measured in parallel experiments and the pitting index, $PI^{(23)}$ defined as the ratio of $\text{std}(i)$ to average galvanic current of carbon steel in 1M NaCl solution. The fluctuation in R_n and R_p are similar, the former being higher than the later by an order of magnitude. The values of pitting index ranged between 0.01 to 0.02. Figure 5 shows the variation of noise resistance, polarization resistance, and pitting Index of carbon steel with time in 0.01 M NaNO_2 + 1M NaCl. The R_n was high in 0.01 M NaNO_2 solution and decreased with addition of NaCl, finally settling at values higher than that in 1 M NaCl alone. R_p also followed a similar trend. PI was in the range of 0.1 - 0.6.

Figure 6 shows typical potential - time records and their power spectrum of carbon steel in 1M NaCl and 0.01 M NaNO_2 solutions. The power spectrum in the two solutions were not significantly different from each other. Figure 7 shows typical current - time records and their power spectrum of carbon steel in 1M

NaCl and 0.01 M NaNO₂ solutions. It can be seen that the galvanic current was two order of magnitude lower in the sodium nitrite solution compared to that in sodium chloride solution. The power of the current noise was also less in the nitrite solution. Unlike the potential noise spectrum, the current noise spectrum as well as the galvanic current was different in the two solutions. Both the potential and the current noise spectrum showed characteristics of white noise. Figure 8 shows the average noise power of current noise between 0.01 Hz and 0.25 Hz. with time. The average power was calculated as the arithmetic mean of noise power between the specified frequencies. The average power decreased drastically with time in 0.01 M NaNO₂ solution. Addition of sodium chloride increased the average power bringing it close to that in 1M NaCl solution but remained less than that. The corrosion rate measured by polarization is shown in figure 9. Lower corrosion rate in presence of sodium nitrite is very evident. The behaviour of corrosion rate and average power of current with time is very similar. It clearly shows that sodium nitrite inhibits the corrosion of carbon steel, although the inhibition efficiency was low. The overall inhibition efficiency from weight loss measurement was 45 % in ten days immersion time.

2. ALUMINIUM

The open circuit potential of Aluminium 1S varied between -700 mv and -1000 mv in 0.01 M, 0.1 M and 1M NaCl solutions. Initial values were -827mv, -690 mv - 838 mv in 0.01 M, 0.1M and 1M solutions and within 24 hours, it reached -900 mV to -950 mV in each solution. Once the initial fluctuation was over, it became steady for the rest of the immersion period. The fluctuation in the potential and the galvanic current is shown in figure 10. After the initial fluctuation, the std(v) settled to lower values: < 3 mv in tap water, 4-6 mv in 0.01 M NaCl and 4-15 mv in 1 M NaCl solutions. The fluctuation in current, std(i)

was more severe : Initial $0.03 \mu\text{A}/\text{cm}^2$ to $0.003 \mu\text{A}/\text{cm}^2$ in tap water; $0.06 \mu\text{A}/\text{cm}^2$ to $0.01 \mu\text{A}/\text{cm}^2$ in 0.01 NaCl; $0.13 \mu\text{A}/\text{cm}^2$ to $0.07 \mu\text{A}/\text{cm}^2$ in 0.1 M NaCl and $0.1 \mu\text{A}/\text{cm}^2$ to $0.07 - 0.1 \mu\text{A}/\text{cm}^2$ in 1 M NaCl.

Figure 11 presents the average galvanic current from aluminium in various solutions. In tap water its magnitude was low at 0.4 to $0.57 \mu\text{A}/\text{cm}^2$. In 0.01M NaCl solution, it was 0.7 to $2.48 \mu\text{A}/\text{cm}^2$. In 0.1 M NaCl solution aluminium showed high initial galvanic current, 5 - $6 \mu\text{A}/\text{cm}^2$ but decreased steadily. Except the initial period, galvanic current was highest in 1M NaCl solution. Figure 12 shows the variation in noise resistance, R_n , polarization resistance, R_p and pitting index, PI of Aluminium in sodium chloride solutions. The range of R_n was 10^3 to 3×10^8 ohms cm^2 . R_p also varied in a similar fashion. Magnitude of R_p decreased with increase in salt concentration. Average power density of current noise between frequency range 0.01 Hz - 0.25 Hz is plotted in figure 13. The power spectrum density of current noise was lowest in tap water and increased with increase in concentration in salt solution. Initial high value in all the cases came down to a low steady value. Figure 14 presents a typical potential - time record and its power spectrum density of aluminium in sodium chloride solution. A typical current - time record and its power spectrum is shown in figure 15. In both cases, the presence of flicker noise is indicated. The slope of $\log(\text{psd})$ vs $\log(\text{frequency})$ curve was in the range of -1 to -2 at frequency above 0.01 Hz in most cases.

The noise parameters like $\text{std}(v)$, $\text{std}(i)$, PI, R_n , noise power, galvanic current have followed the trend in the corrosion behaviour well for carbon steel and aluminium well. A sharp decrease in the potential of carbon steel in the early period of immersion indicates fast corrosion during that time. It is reflected in the increase in $\text{std}(v)$, $\text{std}(i)$, galvanic current and decrease in R_n . Oscillation in the potential towards the end of immersion time may be caused by

formation of thick non-protecting film on the surface. Early inhibition of corrosion of carbon steel by NaNO_2 is shown by increase in potential and decrease in potential fluctuation and current fluctuation, power spectrum density of current noise and increase in R_n . Addition of NaCl caused drastic reversal in these values. That NaNO_2 still inhibited the corrosion process on carbon steel is seen in value of these parameters being between that in NaNO_2 alone and NaCl alone. Corrosivity of solutions like tap water, 0.01 M, 0.1M and 1M sodium chloride with respect to aluminium was reflected well in the noise parameters.

Larger fluctuation in potential and current together is attributed to general corrosion⁽⁶⁾. This was true for carbon steel in NaCl . Potential fluctuation has not been very consistent with actual corrosion rate⁽⁶⁾. In the present case too this was visible. On the other hand, fluctuation in the galvanic current and the mean galvanic current varied in accordance with corrosivity of the medium. Current noise thus appears to be more useful for the purpose of monitoring corrosion rate. Potential noise on the other hand could be more useful for the study of passivity and its breakdown⁽⁵⁾. Theoretical basis for the noise resistance has been discussed by Bierwagen⁽¹³⁾. In our experiments R_n was found to follow the polarization resistance closely but was an order of magnitude higher than that. It means that the corrosion rate calculated with R_n will be less than the corrosion rate calculated with R_p . Corrosion rate calculated with R_p , on the other hand was 5 - 7 times higher than that given by weight loss experiments. In case of aluminium in sodium chloride solutions there was difficulty in measuring polarization resistance with linear polarization due to current oscillation in potential range just anodic to the open circuit potential. R_n appears to be a good indicator of corrosion rate and may be used for corrosion monitoring on-line even if not for absolute corrosion rate. The pitting index was not consistent with the type of attack. It had high values even in those cases where corrosion was of general nature.

Eden's criteria of PI : 0.001 - 0.01 for uniform corrosion, 0.01-0.1 for mixed attack and 0.1 - 1.0 for pitting attack does not have general applicability.

Noise resistance and average power of the current noise appear to be more useful for corrosion rate measurements. Noise resistance may be used in the same way as the polarization resistance. Average power of the current noise may be of direct use. In case of carbon steel, the corrosion current in $\mu\text{A}/\text{cm}^2$ may be related to the power of galvanic current noise by

$$1 \mu\text{A}/\text{cm}^2 \approx 10 \times (\text{psd (I), (A}/\text{cm}^2)^2)^{0.5}$$

Spectrum analysis of the potential noise and the current noise is being treated separately. The present study shows that the average power of the current noise is very close to the corrosion rate. The limits of the frequency must be chosen carefully. While the localized attacks are expected to give rise to low frequency signal, the signal from general attack may be more important at the higher frequencies. It has been found that the frequency range of 0.01 - 0.25 is adequate for this purpose in the present case. Sampling rate in the range 0.1 Hz to 10 Hz did not affect the results. Carbon steel in 1 M NaCl and 0.01M $\text{NaNO}_2 + 1\text{M NaCl}$ gave rise to white noise type of spectrum for both the potential and the current noise; current spectra having higher discriminating power. Aluminium on the other hand gave rise to two different types of spectra: flicker type ($\text{psd} \propto \text{frequency}^{-\alpha}$) in the first 24 hours and white noise type in the later period of immersion. When the corrosion attack was of pitting type the spectrum had a slope of -1 to -2. It shows that the attack on aluminium is pitting type in the beginning and becomes general with very low rate equal to a passive system. Since the specimens are not under any external influence of potential or current, and are not disturbed during the measurements. The galvanic current and its fluctuation can be measured

accurately to a very low level. That makes the noise measurements very useful for monitoring corrosion rates, passivity, coating integrity in on-line systems.

CONCLUSION

It has been shown that the noise measurement is useful for corrosion monitoring. As no external potential or current is imposed on the specimen, it corrodes naturally and in undisturbed manner. Corrosion rate is reflected in standard deviation of potential, standard deviation of current, galvanic current, noise resistance and power of the potential and current noise. The current noise is better suited for distinguishing between corrosivity of the medium. The type of attack is reflected in the shape of the power spectra. General corrosion gave rise to white noise type of spectra while flicker noise type of spectra resulted from pitting attacks.

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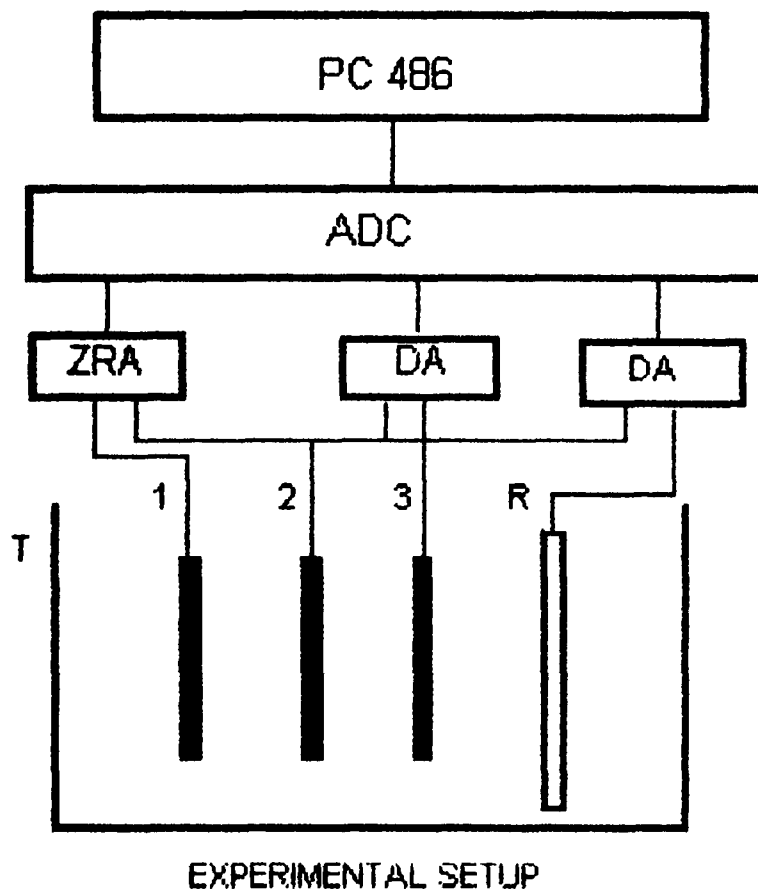


Figure 1. The experimental setup for noise measurement ; 1,2,3 specimen electrodes, R saturated calomel electrode, T tank, DA high impedance differential amplifiers, ZRA zero resistance ammeter and ADC 16 channel A / D converter.

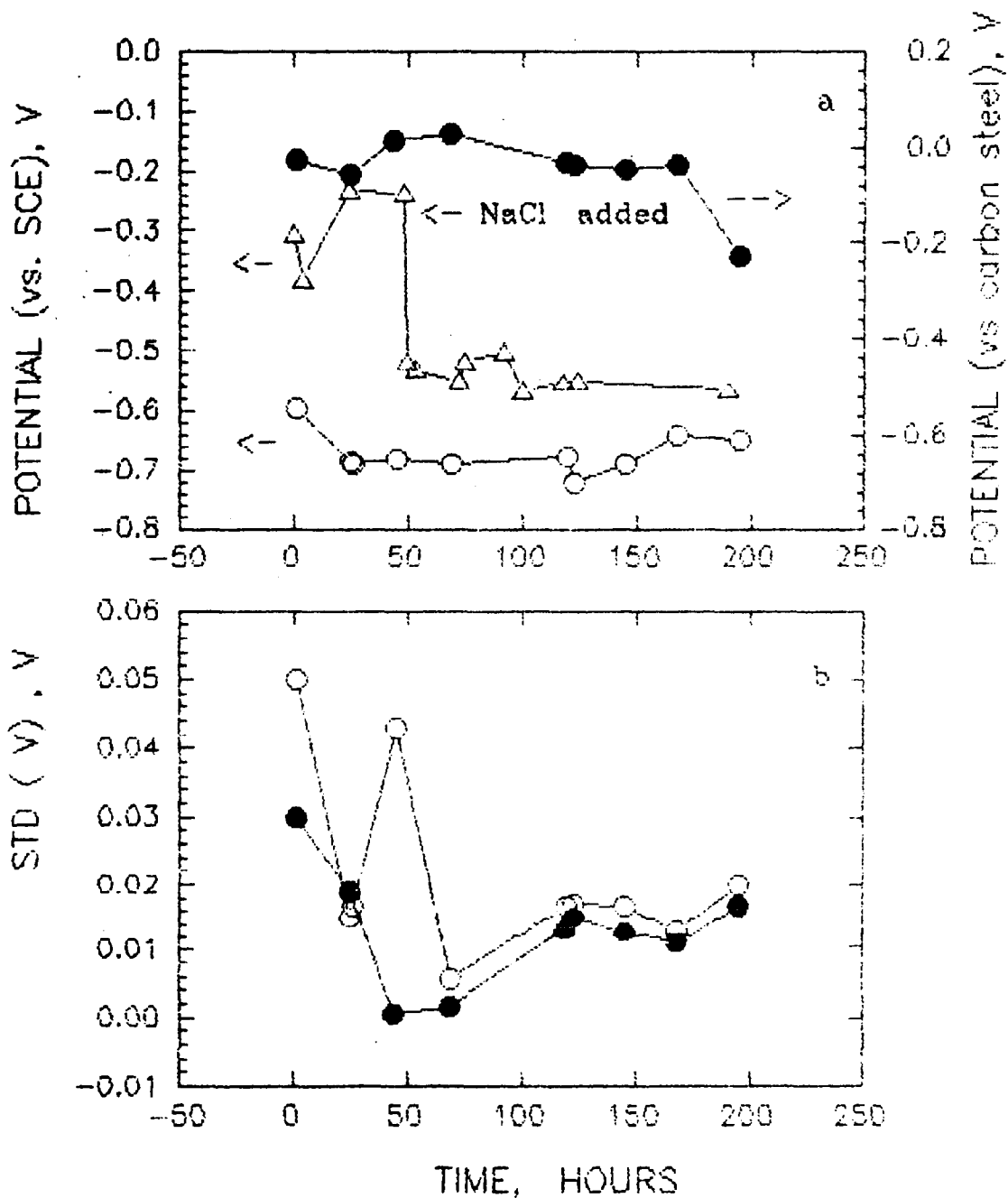


Figure 2. Variation in potential (a) and fluctuation in potential, $\text{std}(v)$ (b) of carbon steel with time in 1M NaCl. ○ - with respect to SCE, ● - with respect to pseudo reference (carbon steel) electrode. Δ - Potential (vs SCE) of carbon steel in 0.01 M NaNO_2 + 1M NaCl.

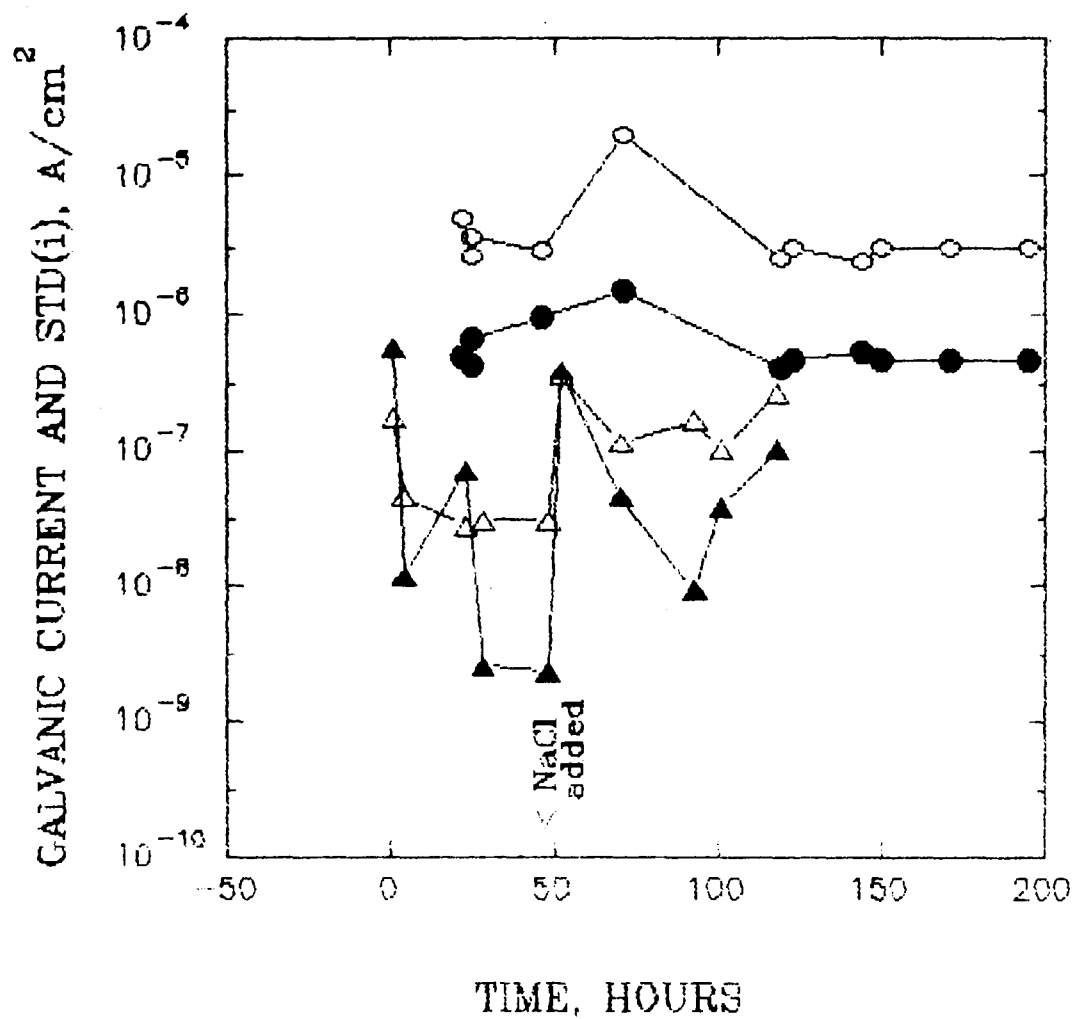


Figure 3. Variation in average galvanic current and standard deviation in current, $std(i)$ of carbon steel in NaCl solution: \circ -galvanic current, \bullet - $std(i)$ in 1M NaCl ; Δ -galvanic current and \blacktriangle - $std(i)$ in 0.01 M $NaNO_2$ + 1 M NaCl.

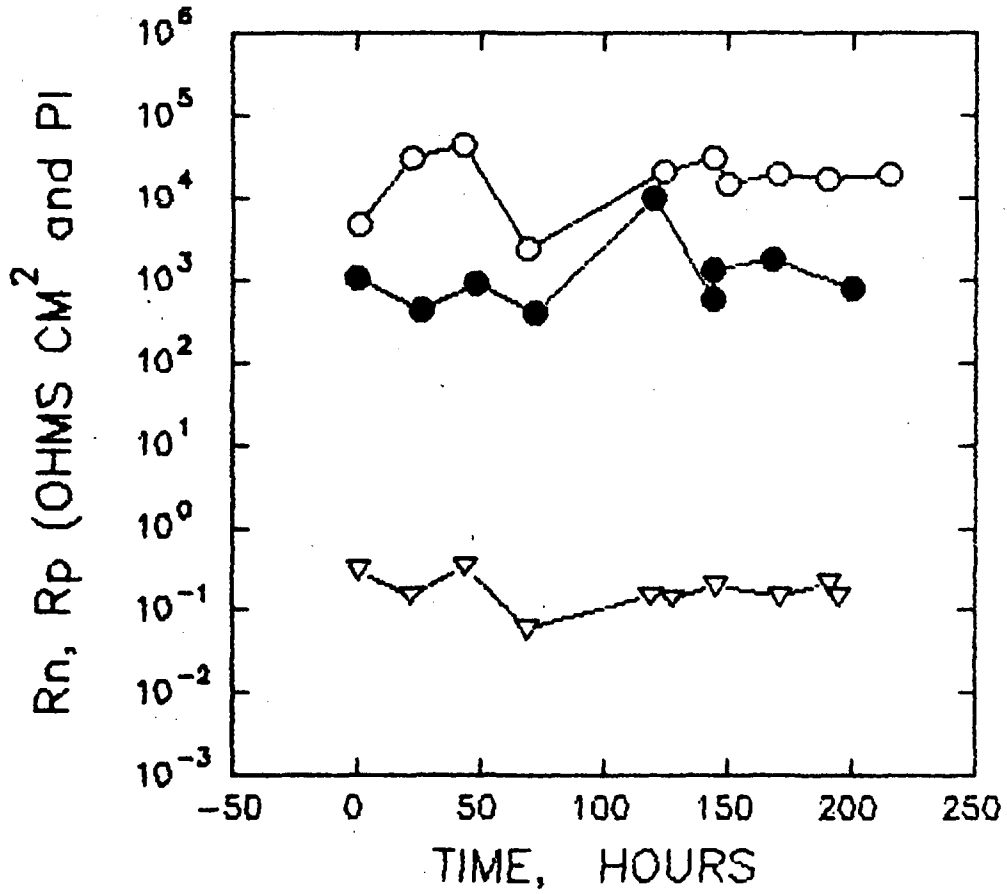


Figure 4. Variation in noise resistance R_n (\circ), polarization resistance R_p (\bullet) and pitting index, PI (∇) of carbon steel with time in 1 M NaCl.

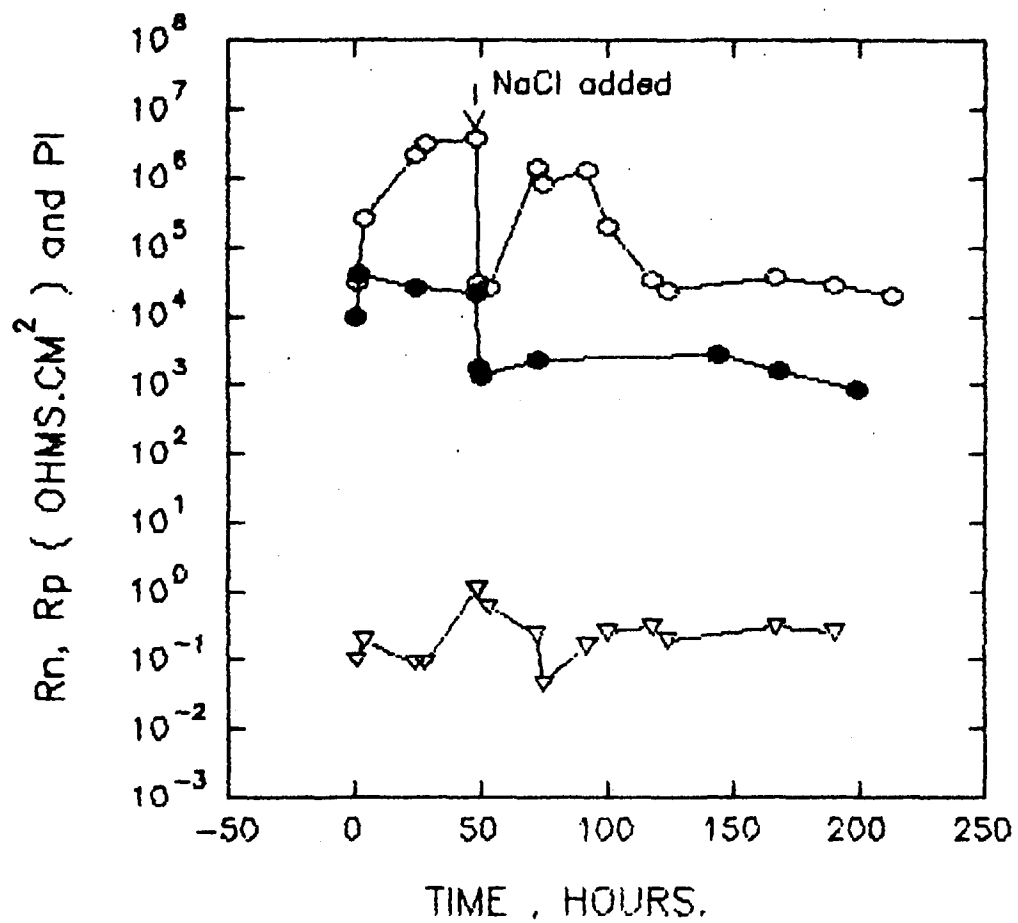


Figure 5. Variation in noise resistance R_n (\circ), Polarization resistance, R_p (\bullet) and pitting index PI (∇), of carbon steel with time 0.01 M NaNO_2 + 1M NaCl .

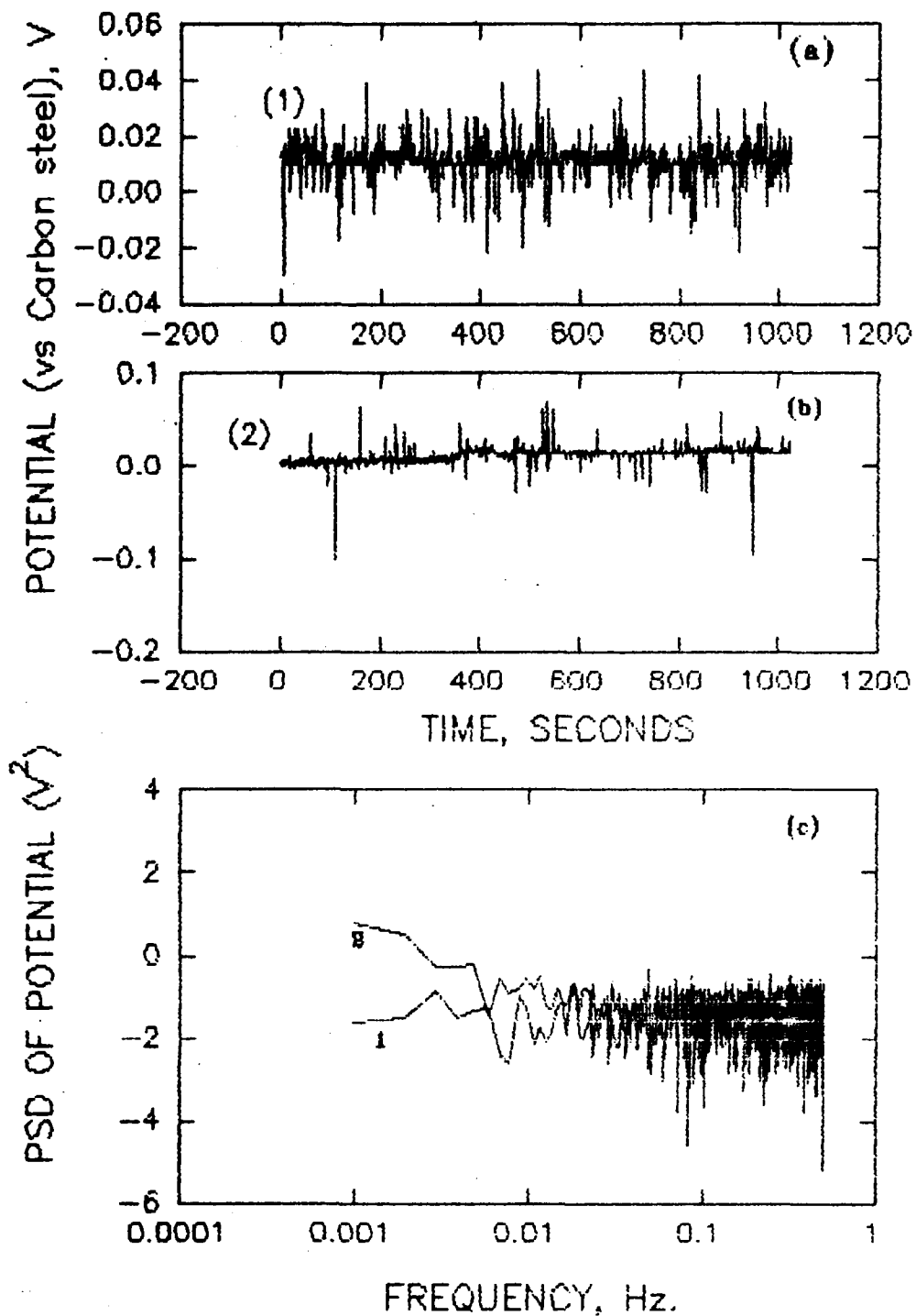


Figure 6. Potential - time records (a,b) and their power spectrum density (c) of carbon steel in (1) 1 M NaCl after 44 hours of immersion, (2) in 0.01 M NaNO_2 after 24 hours of immersion.

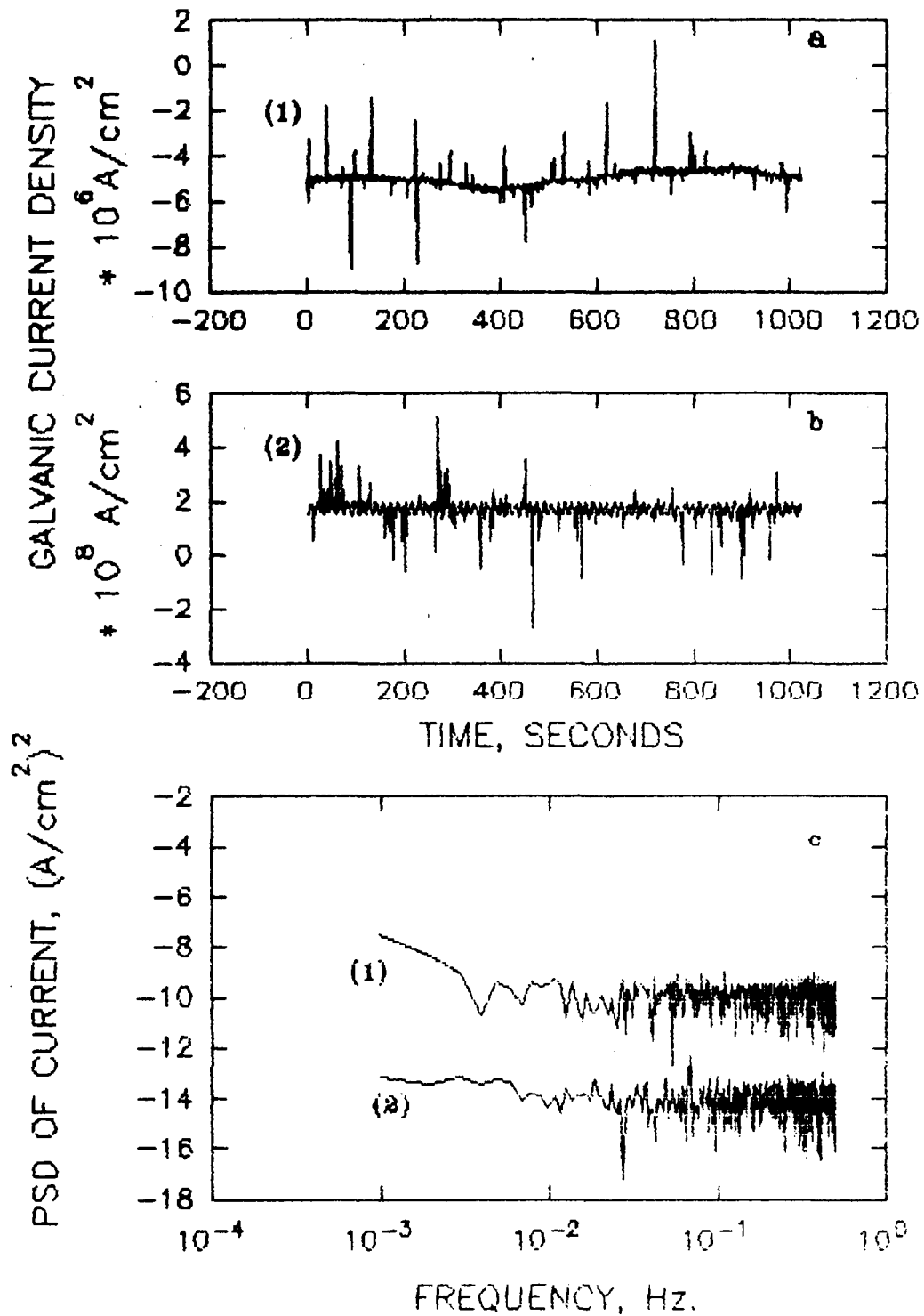


Figure 7. Current - time records (a,b) and their power spectrum density (c) of carbon steel in (1) 1 M NaCl after 22 hours of immersion, (2) 0.01 M NaNO_2 after 23 hours of immersion.

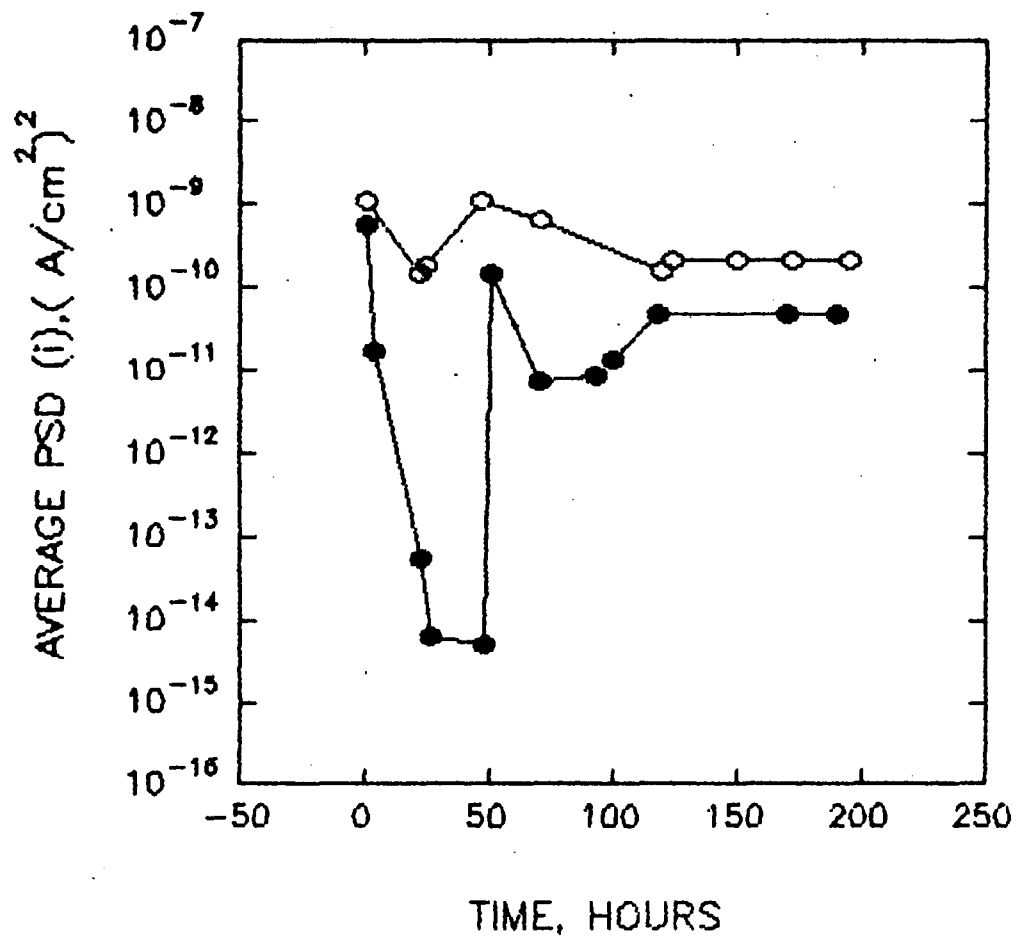


Figure 8. Variation in average power of current noise of carbon steel in ○ - 1 M NaCl and ● - 0.01 M NaNO₂ + 1 M NaCl.

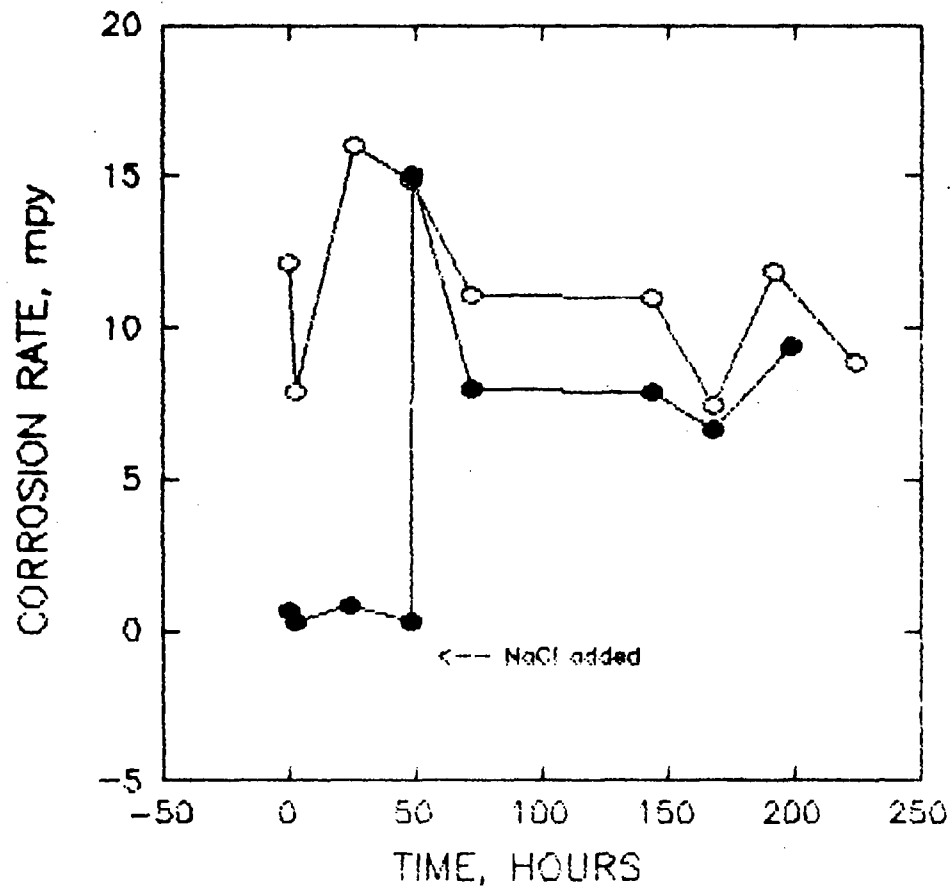


Figure 9. Instantaneous corrosion rate by polarization measurements in ○ - 1 M NaCl and ● - 0.01 M NaNO₂ + 1 M NaCl.

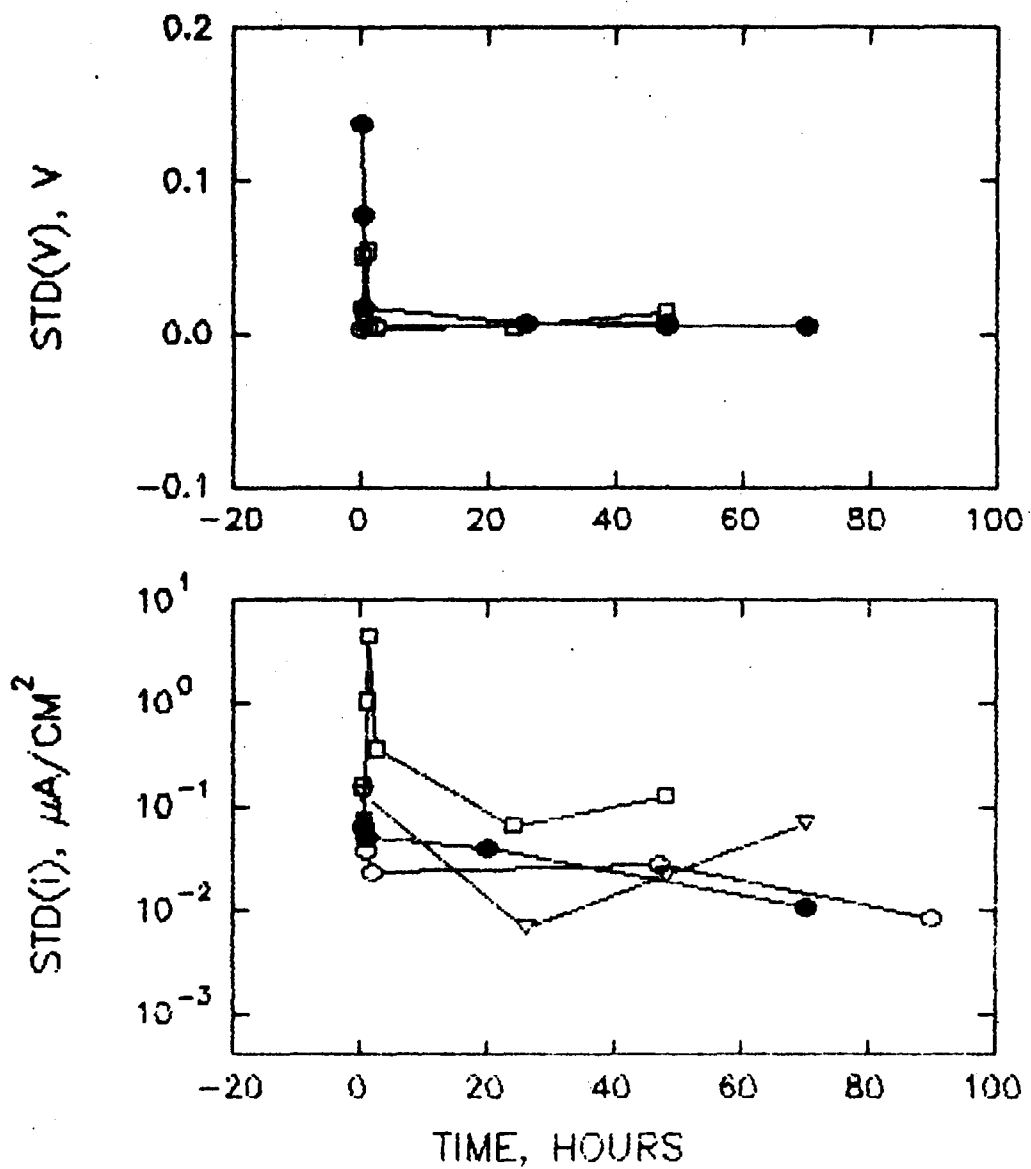


Figure 10. Variation in fluctuation in potential, $std(v)$ and fluctuation in current, $std(i)$ for aluminium with time in NaCl solutions; □ - tap water, ○ - 0.01 M, ● - 0.1 M and ▽ - 1.0 M.

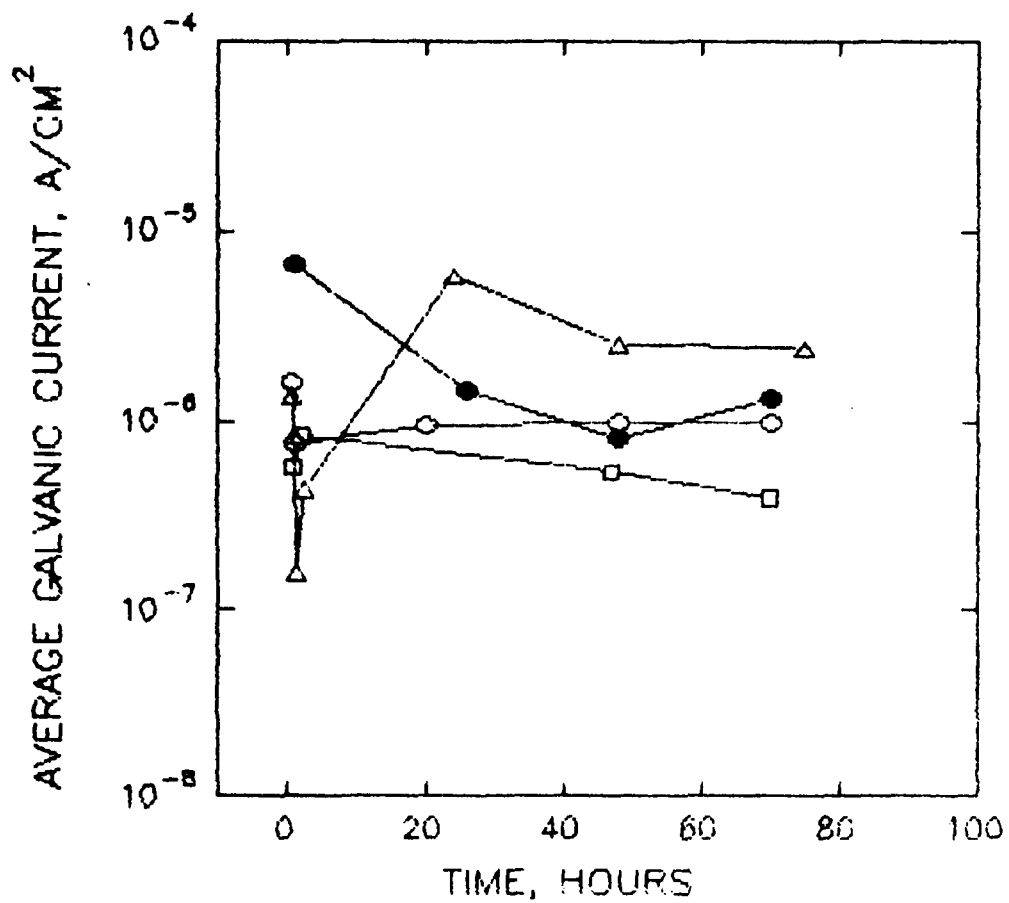


Figure 11. Variation in galvanic current of aluminium in NaCl solutions: □ - tap water, ○ - 0.01 M NaCl, ● - 0.1 M NaCl, △ - 1.0 M NaCl.

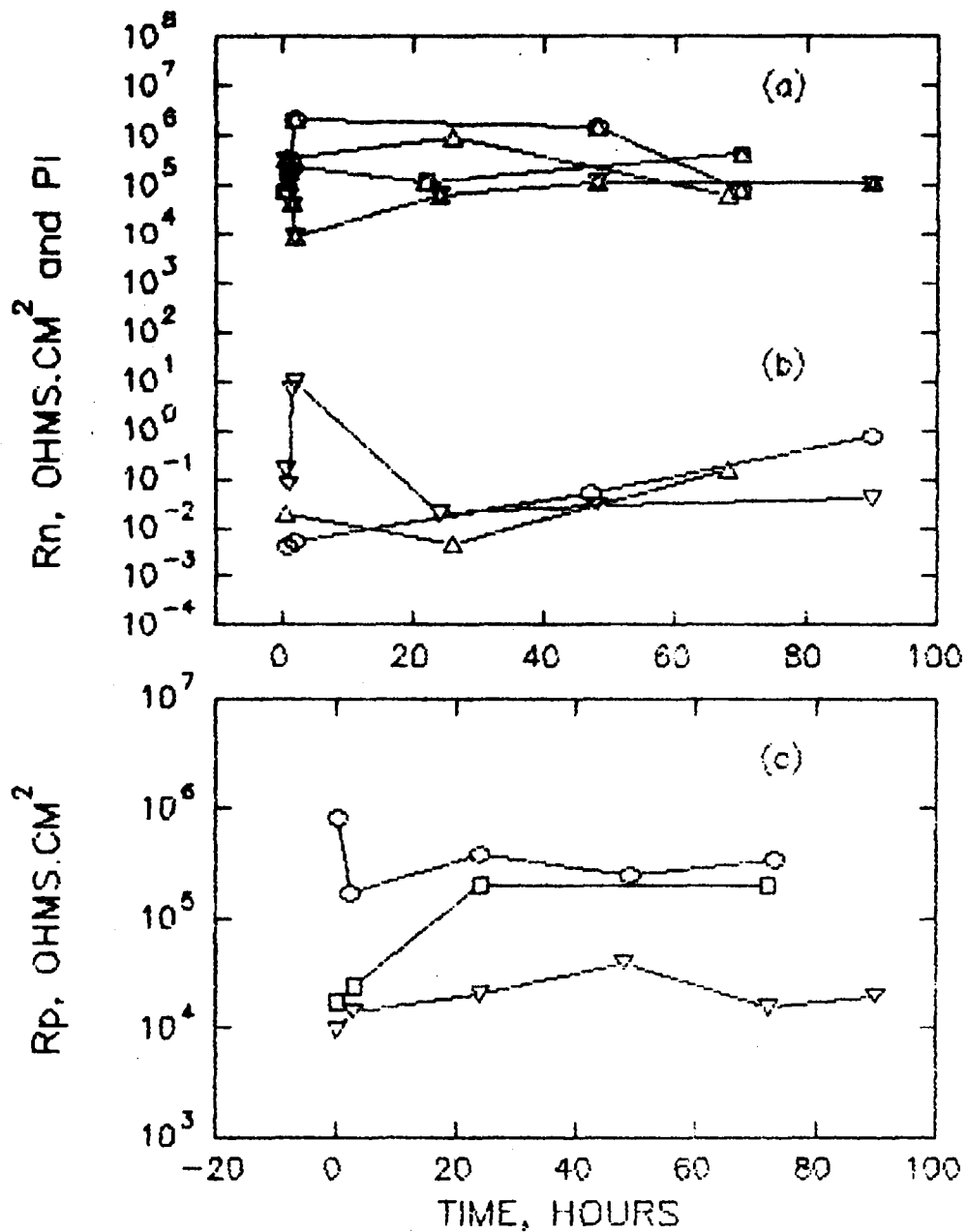


Figure 12. Variation in noise resistance, R_n (a), pitting index, PI (b) and polarization resistance, R_p (c) of aluminium with time in NaCl solutions: O- tap water, \square - 0.01M, Δ - 0.1 M, ∇ - 1M NaCl.

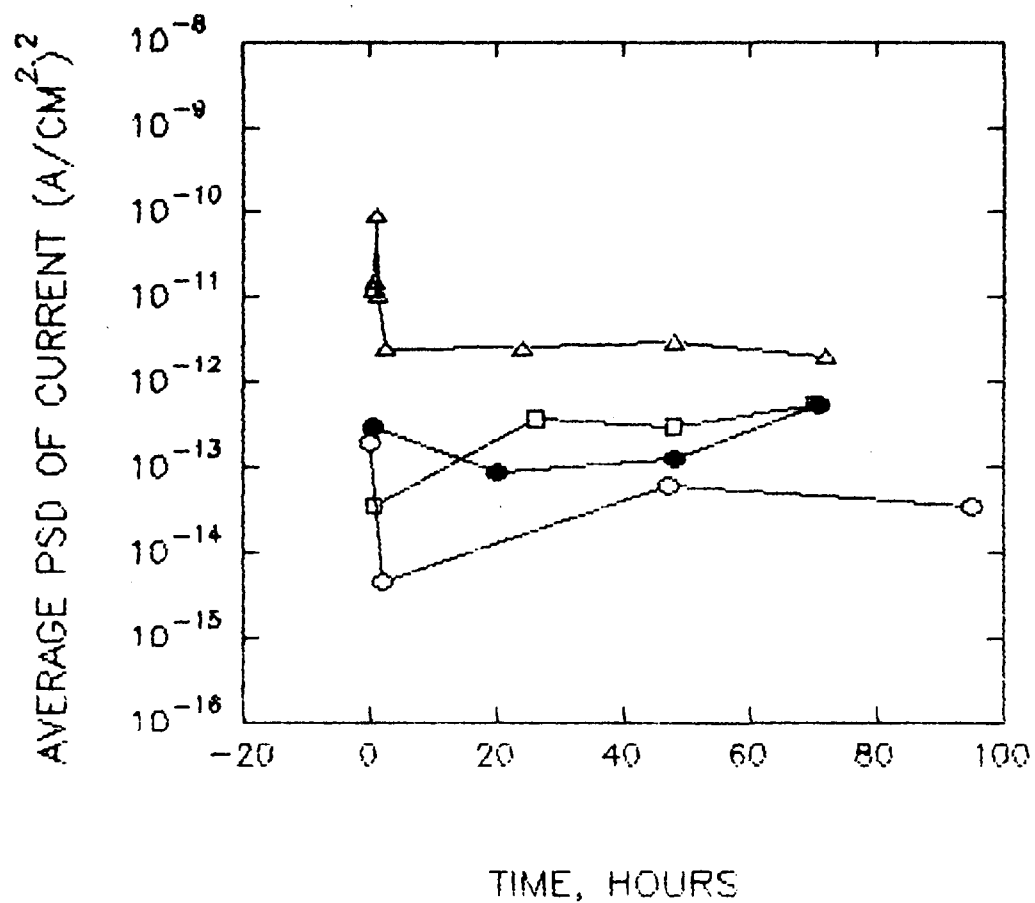


Figure 13. Variation in average power of current noise of aluminium with time, in NaCl solutions; ○ - tap water, ● - 0.01 M, □ - 0.1 M, △ - 1.0 M NaCl.

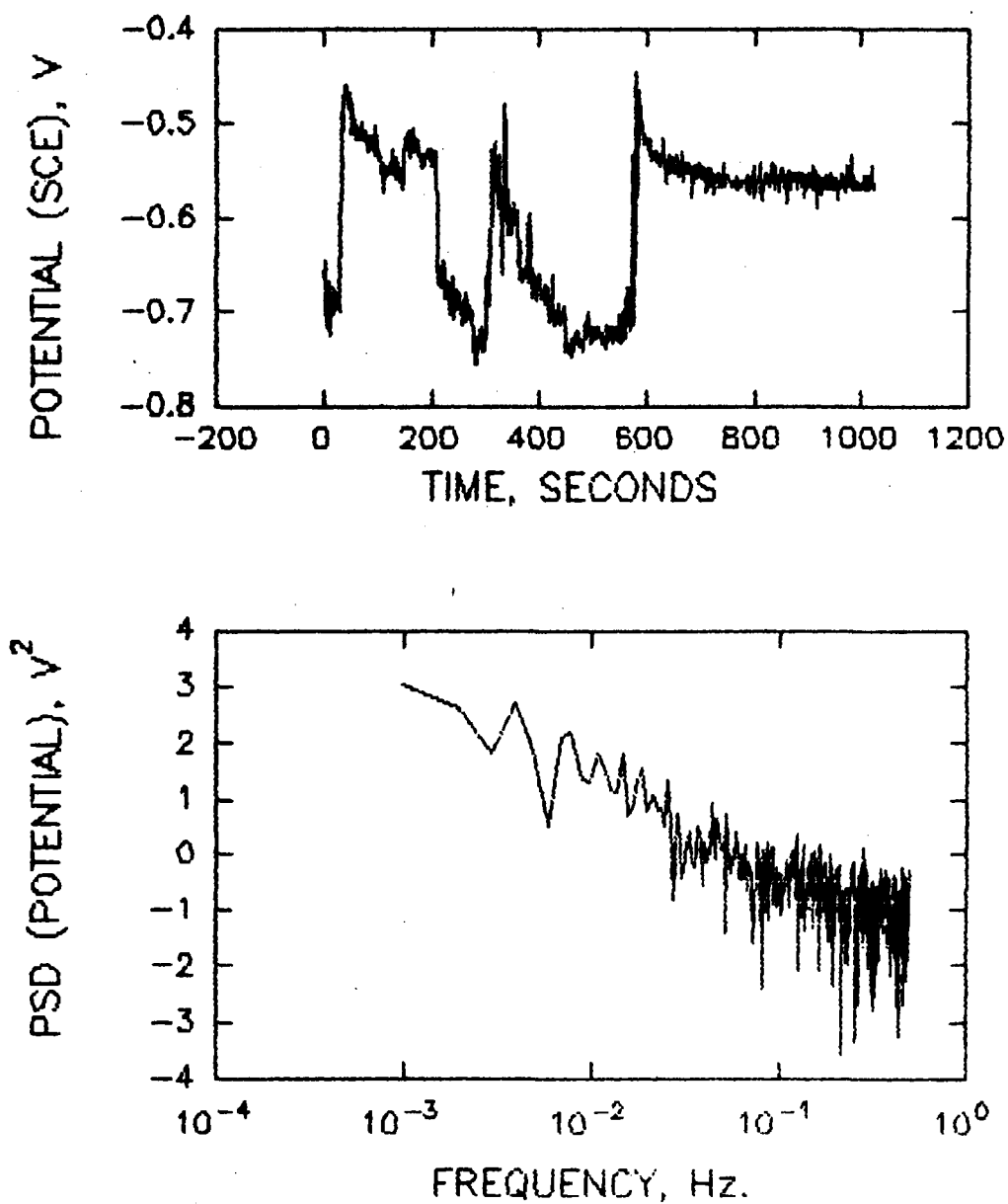


Figure 14. Potential – time record and its power spectrum density of aluminium in 0.1 M NaCl after 20 minutes of immersion.

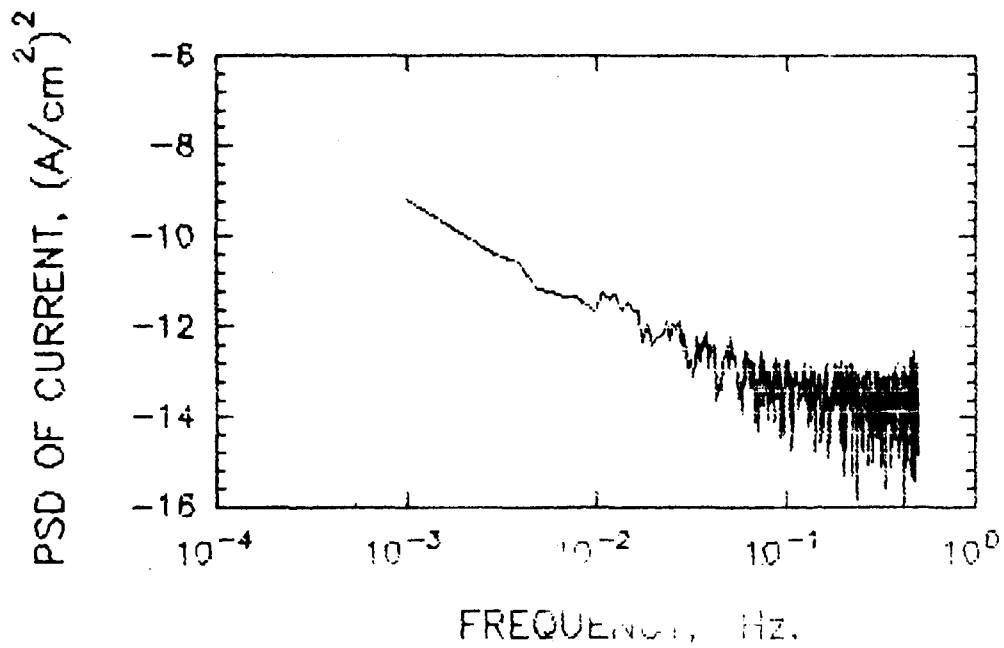
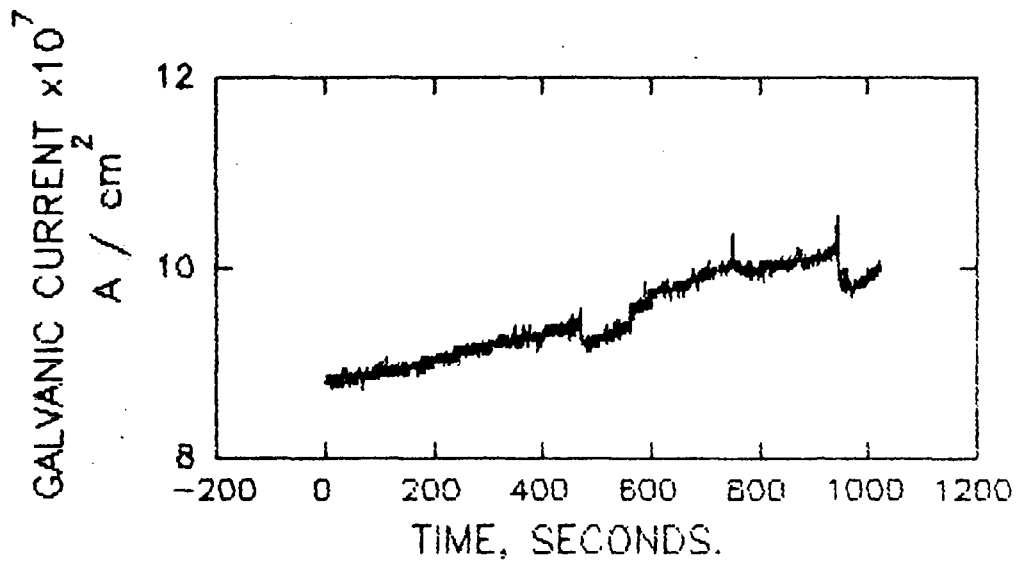


Figure 15. Current – time record and its power spectrum density of aluminium in 0.01 M NaCl after 20 hours of immersion.

