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Corrosion Resistance Behaviour of Duplex (α β) Brass in Nitric Acid Concentrations

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Corrosion resistance behaviour of duplex (α β) brass (65-35% Cu – Zn alloy) was evaluated in different nitric acid and nitric acid - chloride concentrations. The experiments were performed at ambient temperature using potentiodynamic polarization measurement. Various results obtained are presented in the Tables and curve figures. This paper reports the observed electrochemical response of the experimental tests. Different concentrations of the test media were used with and without sodium chloride addition. Tafel and polarization resistance calculations/ measurements were used to estimate the corrosion rate and polarisation resistance of the alloy samples tested. The results obtained showed varied magnitude of corrosion susceptibility of the brass alloy in HNO3 concentrations and its acid chloride test environments.

Keywords: Duplex (α β) brass, corrosion, nitric acid, sodium chloride, potentiodynamic polarization.

1. INTRODUCTION

It is of utmost importance to make appropriate selection of the material to be used for all engineering needs and purposes. This is necessary to ensure safety, long life, reliability, dependability and economic viability of industrial plants, industrial components and other engineering infrastructures/ facilities [1]. Degradation and hence failures of engineering materials in service, particularly metallic alloys, has been majorly due to corrosion [2, 3]. To combat this menace, the selection of the appropriate metal or alloy for a particular corrosion service, is therefore, the most common method of preventing or reducing corrosion damage. Depending upon the environment and/ or where needed, copper alloys of which brasses are unique, are frequently used.

Brass is an alloy of copper and zinc. The proportions of zinc and copper can be varied to create a range of brasses with varying properties which include strength, machinability, ductility, wear resistance, hardness, colour, antimicrobial, electrical and thermal conductivity, and corrosion resistance [4, 5]. Various types of brass respond to corrosion effect in different ways in different environments. Continuous investigation in this regard is therefore an absolute necessity to ensure safe, reliable and functional utilisation and to mitigate ugly application consequences. In this work, duplex (α β) brass was used in nitric acid at different concentrations and also in combination with sodium chloride to simulate both normal and harsh environments.

Nitric acid (HNO_3), also known as aqua fortis and spirit of niter, is a highly corrosive strong mineral acid. Nitric acid is normally considered to be a strong acid at ambient temperatures. There is some disagreement over the value of the acid dissociation constant, though the pK_a value is usually reported as less than −1 [6]. This means that the nitric acid in solution is fully dissociated except in extremely acidic solutions. The pK_a value rises to 1 at a temperature of 250 °C.[6] Since nitric acid has both acidic and basic properties it can undergo an autoprotolysis reaction, similar to the self-ionization of water:

$$2HNO_3 \rightleftharpoons NO_2^+ + NO_3^- + H_2O$$

Nitric acid is a powerful oxidizing agent. The acid reacts with most metals but the details depend on the concentration of the acid and the nature of the metal. Nitric acid can oxidize non-active metals such as copper and silver. With these non-active or less electropositive metals the products depend on temperature and the acid concentration [7].

In its common form nitric acid has a concentration of around 68%, when in a solution containing more than 86%; it is referred to as fuming nitric acid. Depending on the amount of nitrogen dioxide present, fuming nitric acid is further characterized as white fuming or red fuming, at concentrations above 95%.

Whilst available in many concentrations and each offers different properties, the chemical has numerous uses in industry and agriculture [8], the main ones being as: an integral part of explosives manufacture; an agricultural fertilizer; a component of solid rocket fuels, acting as an oxidizer and as a chemical reagent to identify various other metals. It is also used extensively in woodworking to ‘age’ wood and in the jewelry trade nitric acid can be used as an inexpensive process to identify and assess purity of gold, particularly when in low-grade alloys. When in a solution with alcohol and water it is used to etch metals. The *Nitric acid* is commonly used in the food processing and dairy sectors to remove calcium and magnesium deposited during the manufacturing or conversion processes or which may result from continued exposure to hard-water.

In spite of all these, however, the choice of metals and alloys for nitric acid services is quite limited as far as variety is concerned [9] and hence an attempt to seek for a likely inhibition method in a previous study [11]. In service, the acid is very unfriendly to very many alloys. This, accounts mainly for this investigation which may, depending on the outcome of this, be further carried forward in another investigation to include the use of inhibitors. Sodium chloride was used as addition to nitric acid with the hope of further introducing Cl^- (chloride ions) into the test media to make the solutions more aggressive. This could only happen, however, if the reaction chemistry between nitric acid and sodium chloride permits it. It is expected that the outcome of this work will be beneficial to the engineering services and industries.

2. EXPERIMENTAL METHOD

Duplex (α β) brass (65-35% Cu – Zn alloy) samples, in cylindircal form (10mm dia and 10mm long), was used in this investigation. When further analysed, it was confirmed to have these chemical compositions: 0.020% C,0.0025% Si, 0.0046% S, 0.0014% P, <0.0005% Mn, 0.0043% Ni, 0.0006% Cr, 0.0018% Mg, <0.0010% Ag, 0.015% Sb, 0.0035% Bi, 0.0043% As, <0.0005% Sn, 0.020% Co, 0.027% Al, 0.0006% Cd, 35.31% Zn, 0.0041% Pb, 0.0050% Fe, <0.0001% Be, <0.0000% Zr, 0.0060% Au, <0.0005% B, 0.0013% Ti and 64.6% Cu.

The test samples were mounted in turns in araldite resin and connected with a flexible wire connection, ground and polished to fine diamond (1µm), cleaned and rinsed/degreased in an ultrasonic bath using acetone. The samples were immediately kept in a desiccator for subsequent corrosion experimental studies. Potentiodynamic polarisation experiments were performed using each of the flat plate specimens in turns, in which 1 cm^2 surface area of the specimen was exposed to the test solution at room temperature. The experiments were performed using a polarisation cell of three – electrode system consisting of a reference electrode (silver chloride electrode– SCE), a working electrode (WE); and two carbon rod counter electrodes (CE). The potentiodynamic studies were made at a scan rate of 0.00166V/s from -1.5 to +1.5V and the corrosion currents were recorded. The experiments were conducted in eight different concentrations of nitric acid (HNO_3) and nitric acid- chloride, Table 1. The chemicals used: sodium chloride (NaCl) and nitric acid (HNO_3) were prepared from analytic reagent grade (AR).

Table 1. Test Environments

Nitric acid, HNO_3	Nitric acid + 35 g/L NaCl (3.5 %)
3 M	3 M
2 M	2 M
1 M	1 M
0.5 M	0.5 M

The polarisation cell was connected to a potentiostat (Autolab PGSTAT 30 ECO CHIMIE) and interfaced with a computer for data acquisition and analysis. For reproducibility of results, three different experiments were performed for each of the samples under the same conditions; a scan rate of 1 mV/s was maintained throughout the experiment. The experiments were performed in turns in de-aerated condition using nitrogen gas.

3. RESULTS AND DISCUSSION

3.1.3 M HNO_3 and 3 M + 3.5% NaCl test environment

The potentiodynamic polarization curves and table of measurement results for the test specimens recorded in 3 M HNO_3 are presented in Fig. 1 and Table 2 (i) respectively. Fig.1 shows the polarization corrosion curve of the duplex (α β) in 3M HNO_3 alone. The open corrosion potential (OCP), Ecorr was 0.0123389V and 0.012573 V (observed).

Table 2. HNO_3 and HNO_3 + 3.5% NaCl Test Environments Results

S/N	Test environment	ba (V/dec)	bc (V/dec)	Ecorr, Obs (V)	jcorr (A/cm^2)	icorr (A)	Corrosion rate (mm/yr)	Polarization resistance (Ω)	E Begin (V)	E End (V)
(i)	3 M HNO_3	0.037216	0.14161	0.012573	0.000192	0.000602	2.3747	21.256	-0.0351	0.062561
(ii)	3 M HNO3 + 3.5% NaCl	0.021963	0.040276	-0.2129	0.000164	0.000514	2.028	12.003	-0.22797	-0.18158
(iii)	2 M HNO_3	0.071656	0.026124	0.033846	0.000159	0.000499	1.9664	16.675	-0.0058	0.062561
(iv)	2 M HNO3 + 3.5% NaCl	0.014472	0.0333	-0.21862	4.75E-05	0.000149	0.58838	29.365	-0.22797	-0.19379
(v)	1 M HNO_3	0.018552	0.02338	0.04045	0.000136	0.000427	1.6832	10.526	0.02594	0.06012
(vi)	1 M HNO_3 + 3.5% NaCl	0.029478	0.0366	-0.46175	1.91E-05	6.01E-05	0.23692	118.04	-0.23773	-0.18158
(vii)	0.5 M HNO_3	0.012918	0.025561	0.046832	0.000171	0.000538	2.1207	6.9307	0.035706	0.067444
(viii)	0.5 M HNO3 + 3.5% NaCl	0.075915	0.032431	0.012573	0.000192	0.000602	2.3747	21.256	-0.3476	-0.07172

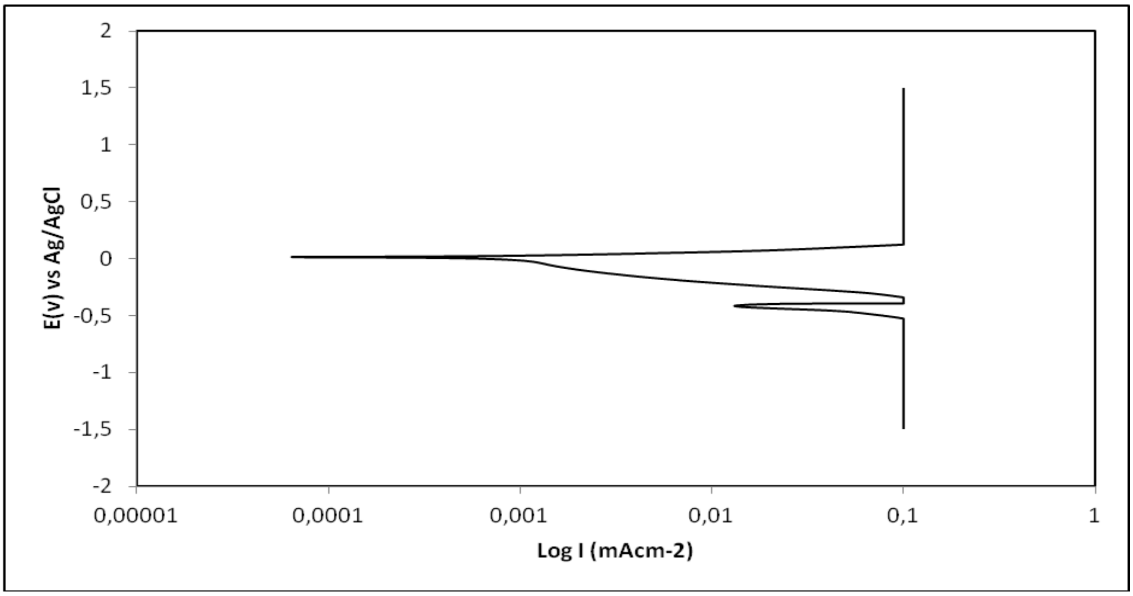

Figure 1. Polarization curve of duplex (α β) brass in 3 M HNO_3

This medium had the highest molarity and hence the most concentrated but without added NaCl. A summary of the result data is presented in Table 2. From the Table, the obtained data values of polarisation resistance, Rρ, of 21.56 **Ω**; corrosion rate of 2.3747 mm/year and current density of 0.000192 (A/cm2), in addition to the OCP values stated above, showed active corrosion reactions that persisted throughout the monitoring period. The very low polarisation resistance and the high corrosion rates values are indications of susceptibility of the tested alloy in the 3 M HNO3.

In the curve, Fig. 1, there was no apparent point to show any value for the passive current density (ipass) or passive potential range, and or also defined point for the primary passive potential (Epp); except that the terminal point of the curve could be used. In this case, the primary passive potential (Epp) value will be 0.118713 V and the critical current density value will be 0.0894073 (A/cm^2). All these values point towards active corrosion reactions and hence no apparent or significant anodic protectibility of the tested alloy in this medium. In general, the corrosion resistance, the passivation characteristics and the anodic protectibility improve as the primary passive potential becomes more active; as the critical current density and the passive current densities decrease, and as the passive potential range becomes broader. Further results here will show if this so for this alloy in nitric acid and nitric acid + NaCl in these test environments.

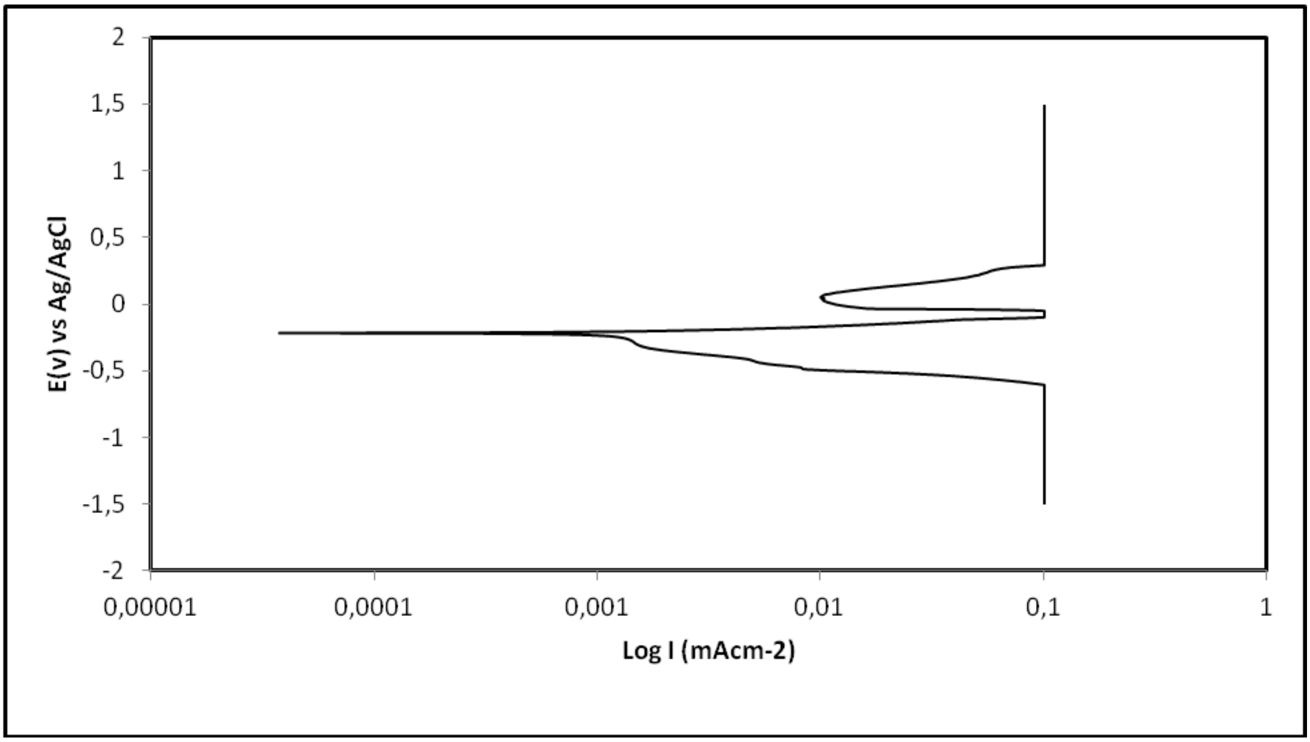

Figure 2. Polarization curve of duplex (α β) brass in 3 M HNO_3 + 3.5% NaCl test environment

In Table 2 (ii) and Fig. 2, are presented, the curve and the Table of the measurement results for the test specimen recorded in 3 M HNO_3 + 3.5% NaCl respectively. In Table 3, various results data values could be obtained. These values include the open corrosion potential, Ecorr, -0.22129 V; and the critical current density, (icorr), 0.000164 (A/cm^2). The others are the corrosion rate, 2.028 (mm/y), and the polarisation resistance, Rρ, 12.003 **Ω**. In comparison with the results in Table 2, it could be seen here that open corrosion potential, Ecorr was more in the active corrosion reactions; the polarisation resistance value was lower and thus indicating more tendency towards corrosion susceptibility. The corrosion rate was still high but a little bit lower than in Table 2(i). There was a slight reduction in current density value. The Cl- (chloride ions) from the NaCl was expected to increase the corrosion magnitude of duplex brass in this test medium at this concentration, but the obtained result did not show that. Though the data values as could be seen above pointed in that direction minimally, the corrosion rate value was reduced. It is difficult to explain this, however, it could be said, plausibly, that nitric acid being a very strong oxidising acid, reacted with the NaCl to give $NaNO_3$, as shown below, [7] which sometimes tend to act like an inhibitor.

$$NaCl + HNO_3 \rightarrow NaNO_3$$

$$\text{The NIE is } NaCl_{(aq)} + H^+_{(aq)} + NO_{3(aq)} \rightarrow NaNO_{3(g)}$$

The results data values from the Table 3, showed that NaCl contributed insignificantly to the active corrosion reactions in this medium

The other results data as obtained from the curve in Fig. 2: the primary passive potential (Epp), -0.0961304 V, critical current density, icorr, 0.0951752 (A/cm^2), and the passive current density, (ipass), 0.0106689 (A/cm^2), confirmed the observation/ results explained above. Addition of NaCl gave a minimal tendency towards active corrosion reactions when compared with the test in the HNO_3 acid alone as presented in Table 2(i) and Fig.1.

3.2. 2 M HNO_3 and 2 M + 3.5% NaCl test environment

The potentiodynamic polarisation measurement results obtained for the tests in 2 M HNO3 and 2 M HNO3 + 3.5% NaCl media are presented in Table 2 (iii-iv) and in Figs. 3 and 4 respectively. In the Table and in Fig. 5, it could be seen that the corrosion rate value, 1.9664 mm/y, decreased when compared with the 3 M HNO_3 test environment. Similarly, the polarisation resistance, Rρ, value, 16.675 **Ω**, increased. The open corrosion potential (Ecorr) with a value of 0.033523 V, was positive and a tendency towards passive corrosion reactions phenomenon. There was also a reduction in the current density value, 0.000159 A/cm^2. All these indicated that this environment behaved less aggressively in corrosions reactions and hence the lower corrosion magnitude as indicated by the data values obtained. The HNO_3 concentration and hence the strength of the acid medium of 2 M which was lower than the 3 M could be associated with this reduced corrosion susceptibility of the alloy in the test environment.

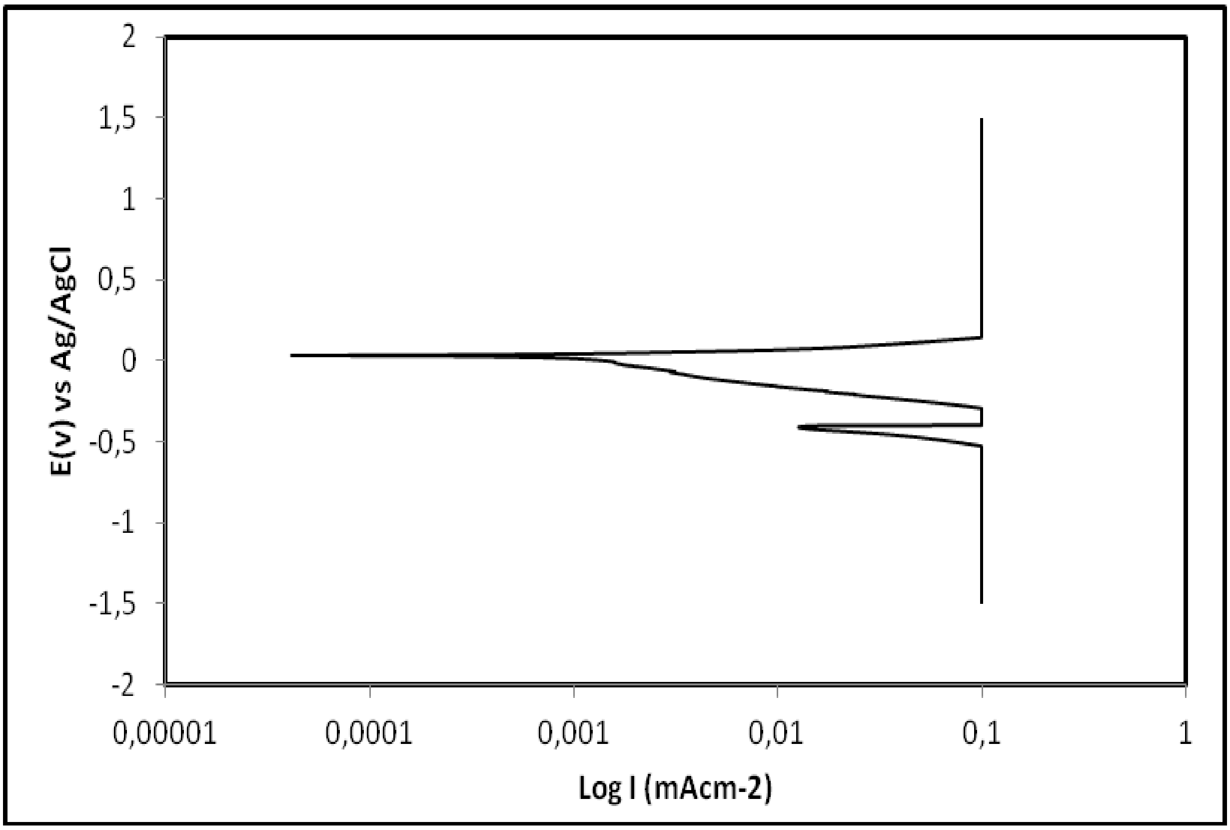

Figure 3. Polarization curve of duplex (α β) brass in 2 M HNO_3

Just like the results in Fig. 2 and Table 2 (ii), the addition of NaCl to the HNO3 test medium with the aim of making the corrosion of the tested alloy more aggressive, did not prove to be, according to the results presented in Fig. 4 and Table 2 (iv). A comparison of the results data values showed the test specimens to be less susceptible to corrosion in this test environment than with the test in 2M HNO_3 alone. The polarisation resistance, Rρ, though still low, recorded a higher value of 29.365Ω. The corrosion rate decreased significantly from 1.9664 to 0.58838 V. Also, the current density decreased from 0.000159 to 0.0000475 A/cm_2; and the Ecorr value, - 0.22492 V increased from 0.033523 V. All these indicate a significant reduction in active corrosion reactions behaviour; and thus confirming that the use of NaCl as acid chloride actually behaved to reduce or stifle the active corrosion reactions of the otherwise very strong nitric acid oxidising environment. Also, just like as in Fig. 2, (with 3 M HNO3 alone), the polarisation curve here became more definitive, as other result parameters emanated from the curve in Fig. 4. The primary passive potential (Epp) recorded a value of -0.0595093 V and the critical current density (icr) value was 0.0889526 A/cm^2.These showed more improvement in the corrosion resistance, the passivation characteristics and the anodic protectibility, that is, less susceptibility of the tested alloy specimen in this test environment. What could cause this corrosion behaviour with particular reference to NaCl influence has been explained in section 3 . 2.

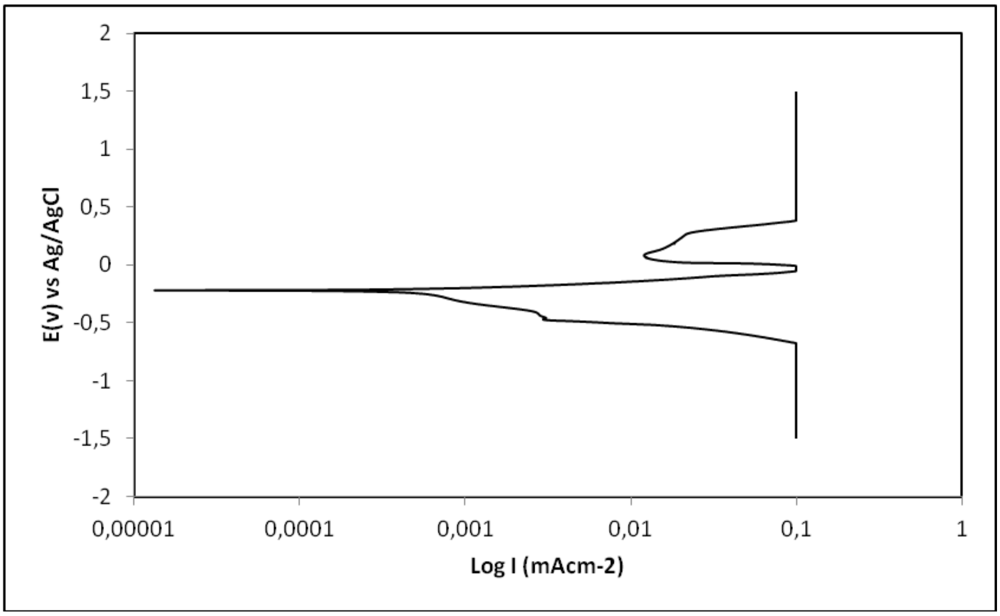

Figure 4. Polarization curve of duplex (α β) brass in 2 M HNO_3 + 3.5% NaCl test environment

3.3. 1 M HNO_3 and 1 M + 3.5% NaCl test environment

Presented in Fig. 5 and Table 2 (v) are the potentiodynamic polarisation measurement results obtained in the experimental tests for the duplex brass alloy specimens in 1 M HNO_3. Also, the results for the tests in HNO_3 + 3.5% NaCl test environment are presented in Fig. 6 and Table 2 (vi).

In spite of the low concentration of HNO_3 in this experiment, the results obtained resembled the previous ones at higher concentrations of the acid. From Table 2 (v), the open corrosion potential, Ecorr, value achieved was 0.037515; the current density, icorr, value was 0.000136; and with a corrosion rate value of 1.6832 V. The polarisation resistance, Rρ, value was 10.526 Ω.

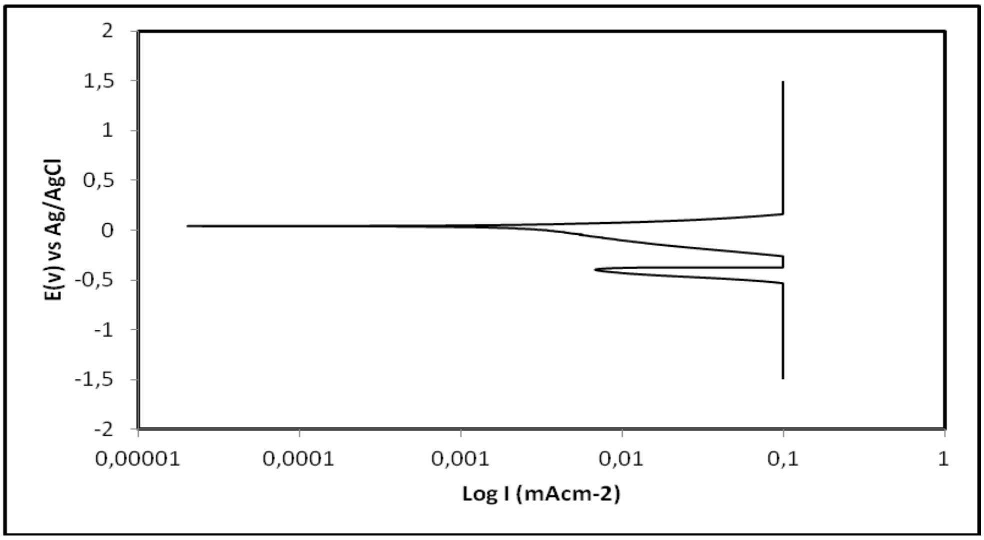

Figure 5. Polarization curve of duplex (α β) brass in 1 M HNO_3 test environment

With this concentration of the acid environment, more than the other previous results with the test in 1 M HNO3, which have been following some particular trends as will be seen in Table 3, the Ecorr value decreased, the current density decreased, the corrosion rate decreased; and the polarisation resistance decreased. Again, it all pointed towards reduced corrosion reactions behaviour and hence lower magnitude of corrosion of the tested metallic alloy in this environment all which are a reflection of the low acid concentration used comparatively. Curiously, the decrease in the polarisation resistance value was an indication of more vulnerability of the metal in this test environment. This can be plausibly, associated with the decreasing oxidising power of the acid medium with the decrease in concentration which tends to decrease the surface resistance of the electrode to corrosion reactions.

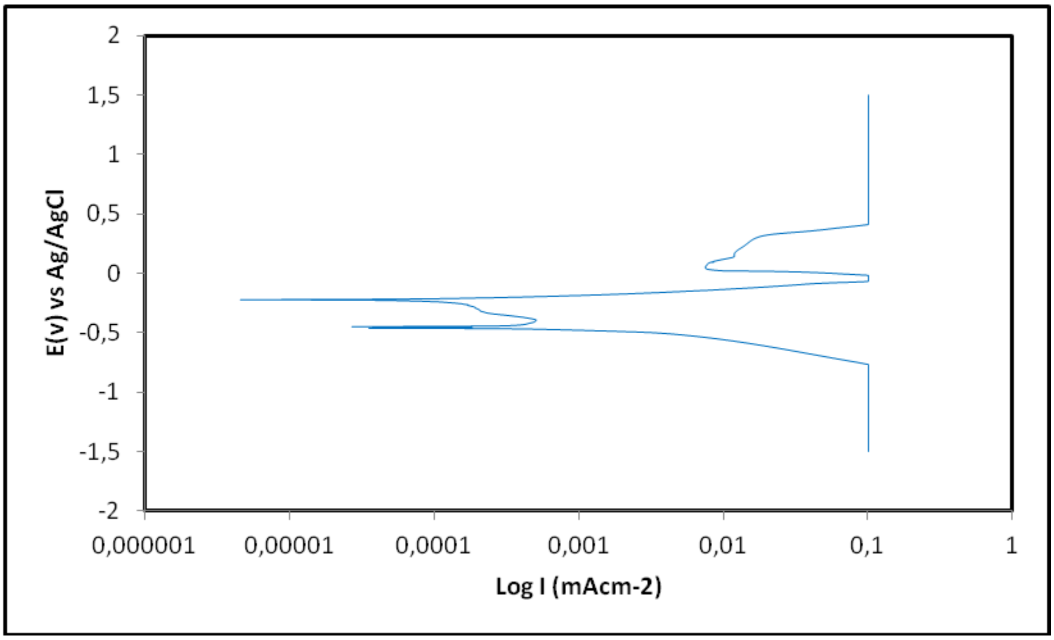

Figure 6. Polarization curve of duplex (α β) brass in 1 M HNO_3 + 3.5% NaCl test environment

The corrosion polarisation curve profile changed when NaCl was added to the acid test environment, and so also the results data values trend. These results are presented in Fig. 6, Tables 2 (vi) and 3 respectively. Compared with Fig. 5 and Table 2(v), the values of open corrosion potential, Ecorr, (-0.2315 V) increased; current density, icorr, (0.0000191 A/cm_2) decreased; corrosion rate (0.23692 V) decreased significantly; and the polarisation resistance also increased significantly. This has been the trend for the test media with the NaCl addition as acid chloride test environment. Duplex (αβ) brass can be described to be very relatively safe in this test environment instead of the anticipated drastic active corrosion reactions behaviour with intense alloy corrosion. From the Fig. 6, the primary passive potential, Epp, value (- 0.0717163 V); and the critical current density, icr, (0.0891632 A/cm_2) value supported the above results of improved passivation characteristics, corrosion resistance and anodic protectibility.

3.4. 0.5 M HNO_3 and 0.5 M + 3.5% NaCl test environment

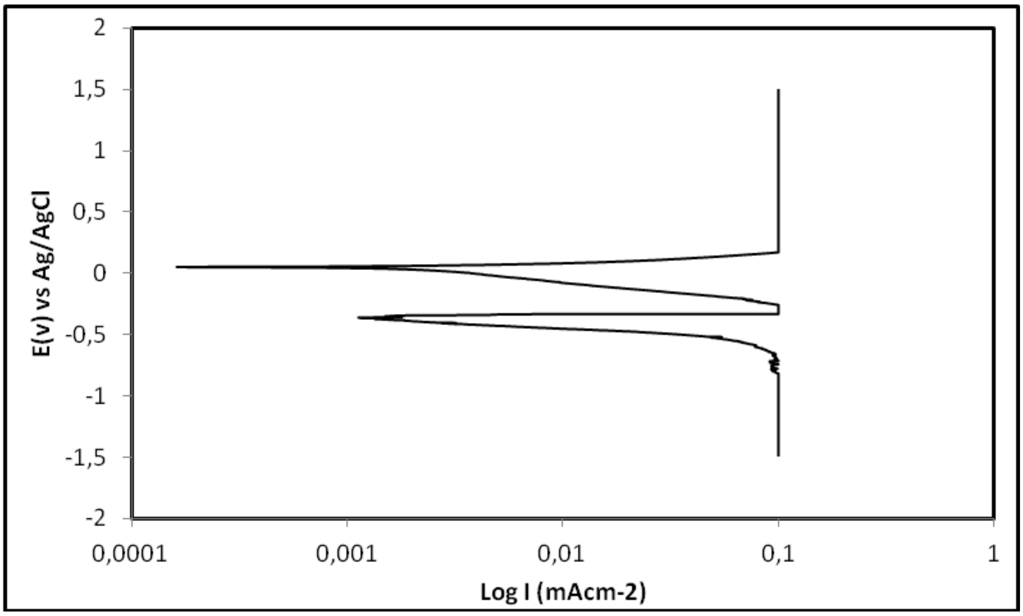

Figure 7. Polarization curve of duplex (α β) brass in 0.5 M HNO_3 test environment

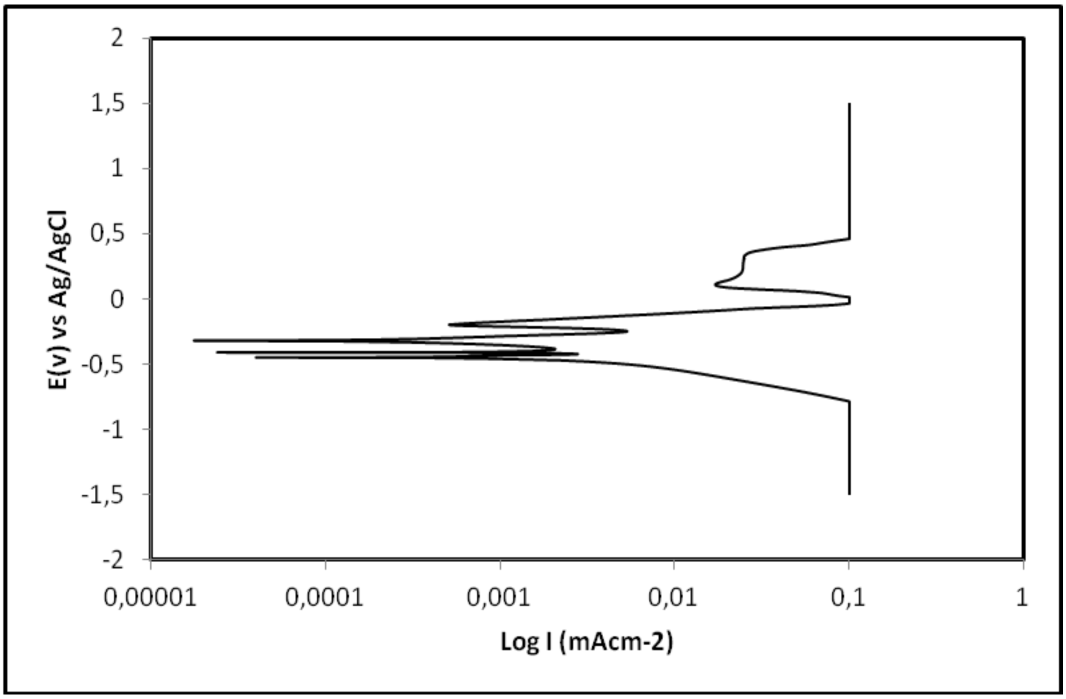

Figure 8. Polarization curve of duplex (α β) brass in 0.5 M HNO_3 + 3.5% NaCl test environment

The results obtained for the corrosion polarisation experiments for the duplex brass test specimens in 0.5 M HNO_3 and 0.5 M HNO_3 + 3.5% NaCl test environments are presented in Table 2(vii) and Fig.7; and Table 2(viii) and Fig. 8 respectively.

From the results of the experiments in 0.5 M HNO_3 test environment alone, Table 2(vii), the Ecorr has the second highest value, 0.041931 V, so far in all the tests performed and reported above. This open corrosion potential value shows passive corrosion reaction behaviour like the others at the beginning of the experiment. The corrosion current density (icorr) value, 0.000171 A/cm2, was also the second highest of all the tests in nitric acid environment alone and only next to the test in 3 M HNO_3 medium. The corrosion rate value, 2.1207 mm/y, followed the same trend; while the corrosion polarisation resistance (Rρ) value, 6.9307 **Ω**, the lowest of all shows the tested alloys to be most susceptible to corrosion in this test medium. This electrochemical corrosion reaction behaviour of this low acid concentration seems difficult to explain. However, this concentration was the lowest used, it was also the one that seemed to permit more dissociation of nitric acid that gave the NO_3^- (nitrate ions) more freedom to initiate corrosion attack on the surface of the tested specimens. This could be the reason for the low corrosion polarisation resistance and hence high corrosion rate of the test electrodes and apparent susceptibility to intense corrosion attack of the alloy. In fact, the effect of nitrate ion is known [10] to accelerate corrosion of brass at low acid concentration. This behaviour was attributed to the adsorption of the ion on the Zn part of the alloy where it enhances corrosion while the ion adsorbed on the Cu part of the alloy leads to the retardation of corrosion and the net effect will be the result of these opposing processes. The primary passive potential, Epp, value, 0.162659 V obtained from Fig. 7 and the critical current density, icr, value, 0.0926544 A/cm^2, all pointed to the test alloy's susceptibility to corrosion in the acid medium at that concentration.

The tests in 0.5 M HNO_3 + 3.5% NaCl test environment, Table 2(viii) and Fig. 8 present a different feature from the test without NaCl (Fig.7 and Table 2 (vii). These results recorded the lowest corrosion rate value, 0.11697 mm/y in the whole of the tests performed. The current density also was the lowest of all in value, 0.00000945 (9.45E- 06); the open circuit corrosion potential, Ecorr, was -0.29958 V. The recorded polarisation resistance, Rρ, value, 332.72 **Ω**, was the highest achieved in the whole of the experiments. All these are indications of good corrosion resistance and not susceptibility in this test medium that contained 3.5% NaCl. In this test also, the primary passive potential, Epp, -0.0448608 V, was more active; and the critical current density, icr, decreased when compared with the same results in Fig 7. These are also, indications of improved passivation characteristics, improved corrosion resistance and anodic protectibility.

3.5. Summary

Table 3 (A and B) is a summary of the major results obtained for the test specimens in all the different test environments re arranged. All tests in HNO_3 alone were grouped together as Table 3(A) and those in HNO_3 + NaCl together as Table 3(B). The trend of corrosion behaviour of the duplex brass alloy are very apparent, more particularly with respect to the use of the acid chloride where the results tend to more passive corrosion reactions and increasingly became protective. The corrosion mechanism, obviously was that of the anodic dissolution of the tested metal alloys by the strong nitric acid medium used.

Table 3. Re-arranged combined results (Extracted from Table 2).

(A) Nitric acid, HNO3, test environment

Test Environment, HNO_3	Ecorr (V)	Icorr (A/cm^2)	Corrosion rate (mm/yr)	Polarisation Resistance (Ω)
3 M	0.12338	0.000192	2.3747	21.256
2 M	0.033523	0.000159	1.9664	16.675
1 M	0.037515	0.000136	1.6832	10.526
0.5 M	0.41931	0.000171	2.1207	6.9307

(B) Nitric acid, HNO3 + 3.5% NaCl test environment

HNO_3 + 3.5% NaCl	Ecorr (V)	Icorr (A/cm^2)	Corrosion Rate (mm/yr)	Polarisation Resistance (Ω)
3 M	-0.22129	0.000164	2.028	12.003
2 M	-0.22392	0.0000475	0.58838	29.365
1 M	-0.2315	0.0000191	0.23692	118.04
0.5 M	-0.29958	0.00000945	0.11697	332.72

The nitrate ions from the acid would have penetrated the surface film to initiate corrosion attack. This could occur more at the zinc phase of the alloy which will be anodic to the copper phase of the alloy matrix.

4. CONCLUSIONS

Some degree of corrosion susceptibility of the tested duplex (α β) brass specimens was exhibited in the nitric acid test environments alone. All the result parameters showed downward reducing trend except for the 0.5 M acid concentration, but with vulnerability to corrosion as shown by the decreasing polarisation resistance values. The use of acid – chloride, that is, the addition of sodium chloride, however, shifted the polarisation behaviour into significantly reduced active corrosion reactions at all the concentrations of the test environment. For all the results parameters of Ecorr, icorr and the corrosion rate, all the values decreased with the decreasing test environment concentration. The polarisation resistance values increased with the decreasing concentrations. They all showed significant decrease in corrosion. At the lowest concentration of 0.5 M HNO3 + NaCl, the alloy gave a fairly good corrosion resistance performance. In all, the addition/use of NaCl was not found deleterious but rather advantageous. The corrosion mechanism was that of the anodic dissolution of the tested alloy. The alloy, duplex brass, should be used with caution in the HNO3 environment that contains no sodium chloride, particularly at the low concentrations.

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