Behavioral Corrosion Mechanism of Cold Worked Mild Carbon Steel
Immersed in Various Acidic Environments
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Abstract

This research work investigated the corrosion rate of cold worked mild carbon steel in Hydrochloric acid, Nitric acid, Acetic acid, Hydrogen tetraoxosulphate (VI) acid, and phosphorus acid, to determine the corrosivity of each of the acids, and susceptibility cold worked mild steel to corrosion in the used environment, using weight loss measurement approach. The coupons were measured prior to immersion and different weight measurement taken at interval of 24 hours, over the period of 168 hours. The difference in weight between the original weight and the weight at each 24 hour interval (weight loss), was estimated and used for calculating the corrosion rates. The results obtained showed that HNO3 is more aggressive in all the five acids considered.

Keywords: Cold work, mild carbon steel, weight loss, corrosion rate, acidic environment.

INTRODUCTION

Mild carbon steel is a vital material in the construction and fabrication industry [1-2]. It is extensively used in chemical and allied industries for the handling of acid, alkalis and salt solutions [3]. The good properties of mild carbon steel include [4-5]: its ultimate strength which is somewhere in the region of 29-30 ton per in², a modulus of elasticity about 13,000-13,500 ton per in², a yield strength of 15-16 tons per in², very ductile, relatively cheapness of production, high mechanical properties, ease of cold working and ability to be hot worked without loss of mechanical properties. Its major disadvantage is that, it requires efficient protection to prevent corrosion [6].

Cold working is a permanent plastic deformation of a metal at a temperature below its recrystallization point or annealing temperature, that is, any form of mechanical deformation processing carried out on a material below its recrystallization temperature. It is low enough to produce strain hardening. It is usually, but not necessary, conducted at room temperature. It is also referred to cold forming or cold forging.

Cold working process involves the alteration of the shape or size of a metal by plastic deformation. Processes include rolling, drawing, pressing, spinning, extruding and heading [9]. It is carried out below the recrystallization point usually at room temperature. Hardness and tensile strength are increased with the degree of cold work, whilst ductility and impact values lowered. The cold rolling and cold drawing of steel significant improves surface finish [10].

During cold working the crystal structure becomes broken up and distorted, and then the materials become strained or work-hardened. Cold working increases the mechanical strength which makes the material harder but more brittle. Its electrical resistivity also increases. However the metal becomes so hard that further attempt at cold working would result in fracture of the material.
In the industry cold working is a common practice. However, a cold work material might be affected in corrosion environment. The aim of this work is to investigate the general corrosion behavior and mechanism on cold worked in various acidic solution environments which include: hydrochloric acid, acetic acid, hydrogen tetraoxosulphate VI acid, and phosphoric acid with concentration varying from 0.5-2.5 mol per dm$^3$ at room temperature and at fixed time intervals. The objective of this work is to determine the rate of corrosion from weight loss measurement.

**MATERIALS AND METHOD**

The mild carbon steel used in this research work was obtained from Vinupet Steel Company, Warri, and Delta State. The elemental percentage composition was carried out at Delta State Company Limited, Ovian-Aladja, and Delta State.

The experiment involved the use of 25 pieces of cold worked mild steel coupons immersed in various environment of acidic solution. The corrosion environment used includes:

- **Hydrochloric acid (HCl)** of molar mass 36.5g, percentage purity 36%, specific gravity 1.17, and relative density of 1.17g/ml or 1170g/L
- **Nitric acid (HNO$_3$)** of molar mass of 63.01g/mol, percentage purity 68%, specific gravity 1.42 and relative density 1420g/L
- **Acetic acid (CH$_3$COOH)** of molar mass 60.05g/mol, percentage purity 99.7%, specific gravity 1.04 and relative density 1420g/L
- **Phosphoric acid (H$_3$SO$_4$)** of molar mass of 98.0g, percentage purity 85%, specific gravity 1.685g/mol or 1685g/L and
- **V. Hydrogen tetraoxosulphate VI acid (H$_2$SO$_4$)** of molar mass 98g/mol, percentage purity 98%, specific gravity 1.305, relative density 1830.5g/L.

The concentrations vary from 0.5 to 2.5 moles per dm$^3$. The method employed for this research was simply the weight loss method, which involve the difference in weight of the coupon before and after exposure to the various corrosion medium at a given time interval and at room temperature. Different tags were used to ensure that the experiment was not mixed-up. To aid the process in observing the corrosion behavior on cold mild steel, the following equipment and apparatus were used:

**Hacksaw**
- This tool enabled the cutting exercise.

**Vice**
- This device aids the positioning of the flat steel bar prior to cutting.

**Drilling Machine**
- This allows for hole to be drilled enabling a hold-point on the coupon.

**pH Paper**
- This enables the acidity and the basicity of the solution to be known.

**Volumetric Flask**
- Used to hold the corrosive solution in which the coupon is immersed.

**Beakers**
- It was used for measuring of the solutions.

**Vernier Calliper**
- It was used to measure the dimensions and thickness of the coupon.

**Nylon Thread**
- This was used to aid the suspension of the coupons in the solutions.

**Mettler PM II Sensitive Balance**
- It was used to measure the variation in weight of the coupon at different stages.

**Coupons Preparation**
- The flat steel bar of about 1500mm x 20mm was cold worked using three-high rolling mill machine to plastically deform the metal into a cylindrical shape, this was manually done.

**Preparation of Coupon to Size**
- The 1500mm x 20mm (flat steel bar) was cut into 50mm x 19.5mm for ease of experiment, producing 25 sets of coupon. This was achieved by clamping the 1500mm x 20mm flat steel bar into a vice and then using hacksaw to cut the desired dimensions. After which a hole of 4mm diameter was drilled on each coupon using an electrically operated drilling machine with 4mm diameter drilling bit. The drilled hole is to aid the suspension of the coupons in different acidic environment using nylon thread. No further preparation or treatment was given to the coupon except being washed in distilled water as shown in figure 1 below.

Fig-1: Steel bar Specimens
Preparation of Standard Corrosion Solution

The various stock solution used for the experiment were prepared using Equation (1) to Equation (3).

Amount of stock solution in g/dm$^3$ = percentage purity x relative density/100

\[ (1) \]

Number of moles = mass of substance in g per dm$^3$/molar mass in g per

\[ (2) \]

After knowing the concentration of the stock solution, the volume of the stock solution required to prepare each of the environment was calculated using the dilution formula as shown in Equation (3).

\[ V_1M_1 = V_2M_2 \quad (3) \]

Where,

- $M_1$ = Concentration of stock solution
- $V_1$ = Volume of stock solution required to make environment.
- $M_2$ = Concentration of environment to be prepared.
- $V_2$ = Volume of environment to be prepared

For HCL acid

Molar mass of HCL = 36.5g/ mol
Percentage purity = 36%
Specific gravity = 1.17

To calculate the amount of the stock solution:

\[
\text{Percentage purity} \times \text{specific gravity} = \frac{36}{100} \times 1.17 = 42.1 \text{g/L}
\]

Amount of stock in mol/dm$^3$ = \( \frac{42.1}{36.5} = 1.17 \text{M} \)

Thus, 36% of hydrochloric acid contains 1.17M of undiluted HCL

For concentration of 0.5MHCL using the dilution formula

\[
V_1 = M_2V_2/M_1
M_1 = 11.53
M_2 = 0.5
V_2 = 200\text{ml}
V_1 = ?
\]

Hence, $V_1 = 0.5 \times 200/11.53 = 8.67\text{ml}$

Then, Volume of distilled water, 200ml – 8.67ml = 191.33ml

Thus, 8.67ml of concentrated HCl acid was dissolved into 19.33ml of distilled water to give an environment of 200ml to 0.5M.

Same procedures were adopted for all reagents used. Table1 shows the volume of the stock solution (acid) required to prepare each concentration of the different environment.

| Concentration (M) | Volume of acids used (cm$^3$) |
|-------------------|-----------------------------|
|                   | H$_2$SO$_4$ | HCl | HNO$_3$ | CH$_3$COOH | H$_3$PO$_4$ |
| 0.5               | 8.67        | 6.53 | 5.79    | 6.84       | 5.46        |
| 1.0               | 17.35       | 13.05 | 11.58   | 13.69      | 10.92       |
| 1.5               | 26.02       | 19.58 | 17.37   | 20.53      | 16.38       |
| 2.0               | 34.69       | 26.11 | 23.16   | 27.38      | 21.85       |
| 2.5               | 43.37       | 32.64 | 28.95   | 34.22      | 37.31       |

Experimental Procedures

After preparing the various stock solutions, fifty (50) sets of clean beakers were tagged with the name of the various acids, with CW, denoting cold worked specimen. Each of five (5) corrosion environment having ten (10) beakers each, five of which is for cold worked specimen, with various concentration ranging from 0.5M to 2.5M. Prior to immersion, the approximate pH value of each solution was taken, using a pH meter, and the value recorded. The specimens were properly washed with distilled water, dried with clean piece of cloth, and then the initial weight was taken, using the Meter PM II Sensitive Digital weigh balance, and then recorded. Total immersion in 200ml test solution of the specimens into the various environment, was aided with the use of a nylon rope tied to a clean to a clean flattened steel rod of about 3mm diameter, placed on top of each beaker, for support as shown in figure 2 below.

RESULTS AND DISCUSSION

The mild steel material elemental composition from the spark test analysis result is as follow; 0.15% C,
shows the weight in grams (g) obtained from the measurement taken at twenty four (24) hours intervals.

Table 2: Weight (g) of Cold Worked Coupons in Hydrochloric Acid (HCL) Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| Wi         | 18.2847 | 18.0496 | 17.8735 | 17.7044 | 18.1717 |
| 24hrs.     | 18.1706 | 17.8851 | 17.6139 | 17.382  | 17.8116 |
| 48hrs.     | 18.0807 | 17.7561 | 17.5497 | 17.297  | 17.605  |
| 72hrs.     | 18.0007 | 17.657  | 17.3255 | 17.0119 | 17.298  |
| 96hrs.     | 17.8962 | 17.4087 | 17.0379 | 16.7324 | 16.7754 |
| 120hrs.    | 17.646  | 17.1924 | 16.7221 | 16.0795 | 16.3787 |
| 144hrs.    | 17.5485 | 16.9149 | 16.4184 | 15.7005 | 15.8943 |
| 168hrs.    | 17.4509 | 16.7374 | 16.2147 | 15.1215 | 15.4009 |

Table 3: Weight (g) of Cold Worked Coupons in Nitric Acid (HNO₃) Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| Wi         | 18.3432 | 18.2542 | 18.2289 | 18.7698 | 18.3904 |
| 24hrs.     | 16.6399 | 14.3821 | 11.654  | 10.7968 | 10.0893 |
| 48hrs.     | 15.9995 | 14.1878 | 11.4681 | 10.2728 | 9.5347  |
| 72hrs.     | 15.7591 | 13.6935 | 11.0822 | 9.9487  | 8.9874  |
| 96hrs.     | 15.427  | 13.5897 | 10.8793 | 9.6246  | 7.9021  |
| 120hrs.    | 15.2713 | 13.4835 | 10.5811 | 9.0362  | 7.423   |
| 144hrs.    | 15.1828 | 12.2852 | 10.3613 | 8.6489  | 7.0237  |
| 168hrs.    | 15.1828 | 12.7869 | 9.5415  | 8.2616  | 6.4244  |

Table 4: Weight (g) of Cold Worked Coupons in Acetic Acid (CH₃COOH) Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| Wi         | 17.6871 | 18.4322 | 18.3568 | 18.1569 | 18.5254 |
| 24hrs.     | 17.6065 | 18.3511 | 18.2683 | 18.0631 | 18.4223 |
| 48hrs.     | 17.5859 | 18.3302 | 18.2409 | 18.0275 | 18.3807 |
| 72hrs.     | 17.5652 | 18.3093 | 18.2235 | 18.0028 | 18.3591 |
| 96hrs.     | 17.5484 | 18.2874 | 18.1879 | 17.9716 | 18.3115 |
| 120hrs.    | 17.5268 | 18.2713 | 18.1707 | 17.9632 | 18.2904 |
| 144hrs.    | 17.5045 | 18.2486 | 18.157  | 17.9384 | 18.273  |
| 168hrs.    | 17.4822 | 18.2258 | 18.1193 | 17.9136 | 18.2455 |

Table 5: Weight (g) of Cold Worked Coupons in phosphoric Acid (H₃PO₄) Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| Wi         | 18.2602 | 18.3466 | 18.0722 | 17.8402 | 18.2308 |
| 24hrs.     | 18.1683 | 18.2322 | 17.9402 | 17.6774 | 18.1198 |
| 48hrs.     | 18.1213 | 18.212  | 17.9168 | 17.662  | 18.0317 |
| 72hrs.     | 18.0743 | 18.1479 | 17.8633 | 17.6265 | 17.9993 |
| 96hrs.     | 18.0123 | 18.081  | 17.7047 | 17.5592 | 17.9396 |
| 120hrs.    | 17.9483 | 18.0021 | 17.7953 | 17.8292 | 17.999  |
| 144hrs.    | 17.9483 | 17.6413 | 18.1707 | 17.4692 | 17.8284 |
| 168hrs.    | 17.8254 | 17.09109| 17.5996 | 17.3367 | 17.5548 |
Table-6: Weight (g) of Cold Worked Coupons in Hydrogen Tetraoxosulphate (VI) Acid (H₂SO₄) Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M | 1.0M | 1.5M | 2.0M | 2.5M |
| Wi         | 18.0903 | 18.159 | 18.7216 | 18.1082 | 18.4389 |
| 24hrs.     | 17.9803 | 17.9899 | 18.5237 | 17.9066 | 18.2186 |
| 48hrs.     | 17.9172 | 17.8895 | 18.4199 | 17.7717 | 18.0330 |
| 72hrs.     | 17.8441 | 17.8291 | 18.276 | 17.6968 | 17.6473 |
| 96hrs.     | 17.7128 | 17.7331 | 18.1399 | 17.4973 | 17.1739 |
| 120hrs.    | 17.6356 | 17.6523 | 18.0939 | 17.1077 | 16.7954 |
| 144hrs.    | 17.5383 | 17.5081 | 17.9546 | 16.8114 | 16.4510 |
| 168hrs.    | 17.4209 | 17.4038 | 17.3252 | 16.5151 | 16.1066 |

From the measured weight presented in Tables 2 to Table 6 above, the weight loss of coupons was calculated which is the difference between the original weight before immersion and the weight after immersion at a given time. Tables 7 to Table 9 represent the weight loss for the various environments.

Table-7: Weight Loss (mg) of Cold Worked Coupons in HCL Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M | 1.0M | 1.5M | 2.0M | 2.5M |
| 24hrs.     | 0.1141 | 0.1645 | 0.2596 | 0.3224 | 0.3601 |
| 48hrs.     | 0.204 | 0.2935 | 0.3238 | 0.4074 | 0.5667 |
| 72hrs.     | 0.284 | 0.3926 | 0.548 | 0.6925 | 0.8734 |
| 96hrs.     | 0.3885 | 0.6409 | 0.8356 | 0.972 | 1.3693 |
| 120hrs.    | 0.6387 | 0.8572 | 1.1514 | 1.6249 | 1.793 |
| 144hrs.    | 0.7362 | 1.1347 | 1.4551 | 2.0039 | 2.2774 |
| 168hrs.    | 0.8338 | 1.1322 | 1.6588 | 2.5829 | 2.7618 |

Table-8: Weight Loss (mg) of Cold Coupons in HNO₃ Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M | 1.0M | 1.5M | 2.0M | 2.5M |
| 24hrs.     | 1.7033 | 3.8721 | 6.5749 | 7.973 | 8.3011 |
| 48hrs.     | 2.3437 | 4.0664 | 6.7608 | 8.497 | 8.8557 |
| 72hrs.     | 2.5841 | 4.5607 | 7.1467 | 8.8211 | 9.403 |
| 96hrs.     | 2.9162 | 4.6645 | 7.3496 | 9.1452 | 10.4883 |
| 120hrs.    | 3.0719 | 4.7707 | 7.6478 | 9.7336 | 10.9674 |
| 144hrs.    | 3.1161 | 4.969 | 7.8676 | 10.1209 | 11.3667 |
| 168hrs.    | 3.6104 | 5.4673 | 8.6874 | 10.5082 | 11.966 |

Table-9: Weight (mg) Loss of Cold Worked Coupons in CH₃COOH Environment

| Time (hrs.) | Concentrations (M) |
|------------|-------------------|
|            | 0.5M | 1.0M | 1.5M | 2.0M | 2.5M |
| 24hrs.     | 0.806 | 0.8011 | 0.0885 | 0.0938 | 0.1031 |
| 48hrs.     | 0.1012 | 0.102 | 0.1159 | 0.1294 | 0.1447 |
| 72hrs.     | 0.1219 | 0.1229 | 0.1333 | 0.1541 | 0.1663 |
| 96hrs.     | 0.1387 | 0.1448 | 0.1689 | 0.1853 | 0.2139 |
| 120hrs.    | 0.1603 | 0.1609 | 0.1816 | 0.1937 | 0.235 |
| 144hrs.    | 0.1826 | 0.1836 | 0.1998 | 0.2185 | 0.2524 |
| 168hrs.    | 0.2049 | 0.2064 | 0.2375 | 0.2433 | 0.2799 |
Table-10: Weight (mg) Loss of Cold Worked Coupons in H₃PO₄ Environment

| Time (hrs.) | Concentrations (M) |
|------------|--------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| 24hrs.     | 0.0919 | 0.1146 | 0.132  | 0.1628 | 0.187  |
| 48hrs.     | 0.1389 | 0.1466 | 0.1554 | 0.1782 | 0.1991 |
| 72hrs.     | 0.1859 | 0.1987 | 0.2089 | 0.2737 | 0.2315 |
| 96hrs.     | 0.2479 | 0.2545 | 0.2769 | 0.289  | 0.2912 |
| 120hrs.    | 0.2603 | 0.2609 | 0.2816 | 0.2937 | 0.335  |
| 144hrs.    | 0.3119 | 0.3445 | 0.3675 | 0.371  | 0.4034 |
| 168hrs.    | 0.4348 | 0.4357 | 0.4726 | 0.5035 | 0.6746 |

Table-11: Weight (mg) Loss of Cold Worked Coupons in H₂SO₄ Environment

| Time (hrs.) | Concentrations (M) |
|------------|--------------------|
|            | 0.5M   | 1.0M   | 1.5M   | 2.0M   | 2.5M   |
| 24hrs.     | 0.1100 | 0.1691 | 0.1979 | 0.2016 | 0.2203 |
| 48hrs.     | 0.1731 | 0.1695 | 0.3017 | 0.3365 | 0.4059 |
| 72hrs.     | 0.2462 | 0.3299 | 0.4456 | 0.4114 | 0.7916 |
| 96hrs.     | 0.3775 | 0.4259 | 0.2817 | 0.6109 | 1.265  |
| 120hrs.    | 0.4547 | 0.5067 | 0.6277 | 1.0005 | 1.6435 |
| 144hrs.    | 0.5520 | 0.6509 | 0.3675 | 1.2968 | 1.9879 |
| 168hrs.    | 0.6694 | 0.7554 | 1.3964 | 1.5931 | 2.3323 |

**Coupon Total Surface Area**

The total surface area of the coupon was calculated using the formula stated below owing to its geometry as shown below:

\[ A = 2(LW + Wt + Lt) \]  

where,

- \( A \) = Total surface area of the coupon,
- \( L \) = Length of Coupon
- \( W \) = Width of coupon
- \( t \) = thickness of coupon, and

The coupons used for the research were of the following dimension:

\( L = 50 \text{millimeter (mm)} = 1.960 \text{ inches (in)} \)

\( W = 19.5 \text{mm} = 0.768 \text{in} \)

\( T = 2.5 \text{mm} = 0.098 \text{in} \)

Therefore;

\[ A = 2(1.960 \times 0.768) + (0.768 \times 0.098) + (1.969 \times 0.098) \]

\[ A = 3.561 \text{ sq. inch} \]

Density of mild steel = 7.85g/cm³

Considering the hot worked coupon in 0.5M HCl environment at 24h, the corrosion rate was calculated as follows

Weight loss = 0.01098g = 109.8mg

Substituting into “CR” formula,

\[ CR = \frac{0.034 \times 109.8}{7.85 \times 3.561 \times 24} = 87.4 \text{ mpy} \]

Figure 1 and Table 12 show the corrosion rates calculated for the various weight loss. The calculated weight loss obtained for cold worked coupons, increases with increasing acid concentration for all five acids as reflected in the normalized graph, indicating accelerated behavior for the metal dissolution. This result is expected because, with increasing acid concentration, both acidity and Cl⁻ ion concentration were increased too. It could be seen that the weight loss of cold HNO₃ and HCl were higher and this was as the results of the acid concentration. The observed increase in corrosion rate of the cold worked coupons can be attributed to segregation of carbon, or nitrogen atoms at imperfection site produced by plastic deformation, and the higher residual stresses, or energy produced in the material by cold working.

Comparing the corrosion rate of the coupons at different environment shown that HNO₃ proved to be more aggressive, followed by HCL, and H₂SO₄ as while CH₃COOH, proved to be the least aggressive acid among the five acids used for the research for both the hot worked and cold worked coupons.
Table-12: Corrosion Rate of Cold Worked Coupons at Different Environment Concentrations of Acid

| Duration (Hrs.) | 0.5M   | 1.0M  | 1.5M  | 2.0M  | 2.5M  |
|-----------------|--------|-------|-------|-------|-------|
| 24              | 286.6  | 136.06| 82.1  | 140.9 | 175.3 |
| 48              | 225.2  | 150.79| 57.6  | 79.2  | 161.5 |
| 72              | 231.7  | 174.59| 44.1  | 74.7  | 210   |
| 96              | 277.8  | 208.71| 42.6  | 57.9  | 251.7 |
| 120             | 285.4  | 249.48| 42.6  | 64.1  | 261.6 |
| 144             | 302.1  | 352.44| 33.5  | 64.9  | 263.7 |
| 168             | 336.8  | 660.73| 31.8  | 76.9  | 265.2 |

CONCLUSIONS

This study investigated the corrosion behavior of mild steels in different acidic environment (HCl, HNO₃, CH₃COOH, H₂SO₄, and H₂SO₄). The results obtained revealed that corrosion rates increased with increased in acid concentration. It was observed that nitric acid (HNO₃) prove to be a more corrosive acid among five acids considered in this research work. This was followed by hydrochloric acid (HCL), hydrogen tetraoxosulphate (VI) acid (H₂SO₄) and phosphoric acid (H₃PO₄). However, acetic acid (CH₃COOH) proved to be the least corrosive acid. Visual inspection of the coupons after immersion in the various acidic solution environments of different concentrations showed general and pitting corrosion, and the latter became more pronounced at higher level of the various acidic solution environment concentrations.

RECOMMENDATION

Based on the corrosion behavior of cold work mild steel in various acidic environments, further research work should be conducted to determine the effect of temperature change on the corrosion rate of cold worked mild steel.

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