Some Non-destructive Testing for Al metal in 0.1N of NaCl and NaOH

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Abstract:
In this work the corrosion behavior of Al metal was studied by using non-destructive testing (NDT), which is a noninvasive technique for determining the integrity of a material.

The ultrasonic waves was used to measure the corrosion which occur by two corrosive medium (0.1N sodium chloride and 0.1N sodium hydroxide) and study the corrosion by weight-loss method and electrochemical method in addition to performance the microscopic inspection for the samples before and after the immersion in the corrosive medium.

Corrosion parameters were interpreted in these media which involve corrosion potential (Ecorr) and corrosion current density (Icorr).

The results indicate that both media was corrosive but the 0.1N NaOH was more corrosive than 0.1N NaCl.

Micro hardness test indicates that, the hardness value of the testing metal is decrease in 0.1N NaOH solution more than 0.1N NaCl solution with longest time of immersion.

Key words: corrosion, Corrosion potential, corrosion current, ultrasonic inspection, micro hardness.

Introduction:
Non-destructive testing (NDT) is a noninvasive technique for determining the integrity of a material, component or structure. Because it allows inspection without interfering with a products final use, NDT provides an excellent balance between quality control and cost-effectiveness.

The main goal of NDT is to predict or assess the performance and service life of a component or a system at various stages of manufacturing and service cycles. NDT is used for quality control of the facilities and products, and for fitness or purpose assessment to evaluate remaining operation life of plant components.

NDT inspection of industrial equipment and engineering structures is important in power generation plants, petroleum and chemical processing industries, and transportation sector. State of the art methodology is applied to assess the current condition, fitness-for-service, and remaining life of equipment. NDT inspection provides basic data helping to develop strategic plans for extending plant life.

The major six NDT methods, which are largely used in routine services to industry, are:-visual inspection, liquid penetrate testing, magnetic particle testing, electromagnetic or eddy current testing, radiography and ultrasonic testing. [1]
Ultrasonic testing (UT) method uses high frequency sound waves to measure geometric and physical properties in materials. Ultrasounds travel in different materials at different velocities. The ultrasound wave will continue to travel through the material at a given velocity and does not return back unless it hits a reflector. Reflector is considered any boundary between two different materials, or a flaw. The ultrasound generator emits waves and in the same position receives reflected sounds (if any). Comparing both signals (emitted and reflected) the position of the defect and its size can be measured. The UT can be used on civil engineering equipments, outside metallic parts, to verify the granulation of road covering or of concrete.


In this work the ultrasonic waves was used to measure the corrosion which occur by two corrosive medium (0.1N NaCl and 0.1N NaOH) and study the corrosion by weigh-loss method and electrochemical method and micro hardness of the material in addition to performance the microscopic inspection for the samples before and after the immersion in the corrosive medium.

**Materials and Methods:**

The corrosion of pure [Al] was studied in 0.1N NaCl and 0.1N NaOH solutions. Weight loss measurements performed by immersion of (2x3cm²) pure Al in test solutions at room temperature. Ultrasonic measurement performed by measure the thickness of Al sheet before and after the immersion in test solutions by the Ultrasonic Kraut Kramer Branson (DM3) from Germany.

The measurement of corrosion behavior by using electrochemical method was performed by using Wenking M Lab Potentiostat from Bank-Elektronik (Germany) at scan rate (2mV/sec). The measure of polarization behavior was performed using glass cell with three electrodes ,working electrode (pure Al), auxiliary electrode (Pt), and reference electrode (SCE).

The performance of inspection by using BEL from Italia was used to know the change in the microstructure of aluminum before and after the solution treatment.

Micro Hardness was performed by using Digital Micro Hardness Tester HVS-1000.

**Corrosion Test:**

Pure Al, was cut into cylinder shape with (1.4cm) diameter, and made into electrode by pressing a copper wire into a hole on one side and then insulating all but one side with an epoxy resin. The open side was polished mechanically to a mirror finish, rinsed by distilled water and stored in desiccators. The electrochemical glass cell was of the usual type with provision for working electrode (Al), auxiliary electrode (Pt electrode), and a Luggin capillary for connection with a saturated calomel electrode (reference electrode SCE). Electrochemical measurements were performed with WINKING M Lab.
Potentiostat from Bank-Elektronik at a scan rate 2 mV.sec\(^{-1}\).

**Polarization Resistance (Rp):**

From the polarization curves behavior of samples it can be get the corrosion potential (Ecorr) and corrosion current density (icorr) by extrapolation method. Another parameter can be calculated from corrosion measurement which is the polarization resistance (Rp) which represents the measure of the resistance of the metal against corrosion in the immersion solution.

The polarization resistance (Rp) can be determined from Stern-Geary equation [6]:

\[
Rp = \left( \frac{dE}{di} \right)_{i=0} = \frac{b_ab_c}{2.303(b_a + b_c)icorr}
\]

Where E is the applied potential (Volt), i is the current density (A . cm\(^{-2}\)) and ba, ba are anodic and cathodic Tafel slopes respectively.

**Hardness:**

Hardness is commonly defined as the resistance of a material to indentation by a harder material with applied load. Hardness can be quantified by depth of indentation of a hard indenter, usually diamond, and loaded perpendicular to planer surface of the material under test.

The measured hardness of any material depends on parameters associated with the test method, indenter geometry and load, Brinell, Vickers, Rockwell, etc., so that hardness is not an intrinsic bulk property, comparable to elastic modulus, yield strength or fracture toughness. In general, the measured hardness varies with applied load and the indenter shape and dimensions, but also with the microstructure and prior history of the material, the environment, and the test temperature [7].

The Vickers hardness can be calculated by using the following equation:

\[
HV = \frac{2P}{D^2 \sin \frac{\alpha}{2}}
\]

In this relation D is the indentation diagonal in millimeters, P is the load in kilograms and \(\alpha\) is the angle of the pyramid, a known constant equal to 136°. With the help of a special conversion table the HV number can then be translated into different hardness scales, such as Rockwell Hardness A,B,C, etc.[8].

**Ultrasonic inspection:**

Ultrasonic testing method uses high frequency sound waves (2.25-30MHZ) to measure geometric and physical properties in materials. High frequency sound waves are introduced into a material and they are reflected back from surfaces or flaws. Reflected sound energy is displayed versus time, and inspector can visualize a cross section of the specimen showing the depth of features that reflect sound.

**Results and Discussion:**

1- **Ultrasonic inspection**

Ultrasonic inspection show the different in thickness of samples before and after immersion in the experimental solutions which indicate that both media was corrosive but the 0.1N NaOH was more corrosive than 0.1N NaCl. The results of this test were show in table (1), (2) and fig. (1).

Where the aluminum corroded in the basic medium to produce (AlO\(_2^−\)) ions according to the Boursbaix diagram while aluminum metal in neutral medium corroded unless form passive layer of Al(OH)\(_3\), Al(OH)\(_3\).3H\(_2\)O and Al\(_2\)O\(_3\). Thus OH\(^−\) ions attack the surface faster than CI\(^−\).
ions and then obtain less thickness in the basic medium.

2- Corrosion behavior

Polarization experiments were started when the rate at which open circuit potential (E_{OCP}) changed was less and more 200mV.

Fig (2) and (3) show the polarization curve for pure Al in the solutions of 0.1N NaOH and 0.1N NaCl respectively. The below section of curve represent the cathodic region, where the reduction of oxygen can occur according to the following reaction:

\[ \text{O}_2 + 4e + 2\text{H}_2\text{O} \rightarrow 4\text{OH}^- \] (At cathode, in both media)

The above sections of curve represent the anodic region where the dissolution of aluminum can occur according to the following reaction in sodium chloride solution:

\[ \text{Al} \rightarrow \text{Al}^{3+} + 3e \] (At anode, basic and neutral media)

And then:

\[ \text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al} (\text{OH})_3 \]

But in sodium hydroxide solution:

\[ \text{Al} + 2 \text{H}_2\text{O} \rightarrow \text{AlO}_2^- + 4 \text{H}^+ + 3e \]

The rate of reaction can calculated from the following equation [2]:

\[ R_{mpy} = 0.13 \cdot i_{corr} \cdot \frac{e}{\rho} \]

Since: \( R_{mpy} \): Rate in mil/year, \( i_{corr} \): Corrosion current density in \( \mu \text{Amper/cm}^2 \).
\( e \): Equivalent weight, \( \rho \): Density 2.7 gm/cm\(^3\) for Al.

The result of corrosion parameters and rate has shown in table (3) includes corrosion potentials(\(E_{corr}\)), corrosion current density(\(i_{corr}\)) and the rate of corrosion, which indicates that, the NaOH solution more corrosive than the NaCl solution for pure Al depending on (\(E_{corr}\)),(\(i_{corr}\)), and (\( R_{mpy} \)).

3- Weight-loss measurement

The result of the change in weight of samples was shown in table (4). The variation of weight with the immersion time was shown in fig (4) for pure Al in 0.1N NaCl, and 0.1N NaOH solutions. The result of weight loss corresponding thickness losses in ultrasonic inspection.

4- Micro hardness measurement

Micro hardness were tested in Micro Hardness Tester HVS-1000, and calculated from the following equation[9]:

\[ Hv = 1.8544 \times \frac{p}{D_{av}^2} \]

Since :-

\( Hv \): Vickers hardness (Kg / mm\(^2\)), \( P \): load projection (Kg), \( D_{av} \): Diameter average.

The result of micro hardness of samples was shown in table (5), (6) which indicates that, the hardness value of the testing metal is decrease in 0.1N NaOH solution more than 0.1N NaCl solution with longest time of immersion. Fig. (5), (6) show the results of this test. These results explain the corrosion occurs in basic medium more than neutral medium.

5- Microscopic inspection

The microstructure of Al before and after the solution treatment were tested by using BEL from Italia. Fig (7) and (8) show the microscopic inspection for pure Al before immersion in NaOH and NaCl solution respectively. While fig (9) and (10) show the microscopic inspection for pure Al after immersion for (20) days in NaOH and NaCl solutions respectively.
Conclusions:
All the results can be concluded as follow:-
1- The ultrasonic inspection (Thickness mm) for overall(20)days take the following sequence:
Thickness (mm) NaOH solution < NaCl solution
2- The corrosion potential (\(E_{\text{corr}}\)) take the following sequence with the different of medium:
\(E_{\text{corr}}\) (mV) NaOH solution > NaCl solution
3- The corrosion current density (\(i_{\text{corr}}\)) take the following sequence:
\(i_{\text{corr}}\) (µA/cm²) NaOH solution > NaCl solution
4- The rate of corrosion (\(R_{\text{mpy}}\)) take the following sequence:
\(R_{\text{mpy}}\) (mil/year) NaOH solution > NaCl solution
5- The weight-loss measurement show decrease in weight (g) for (20) days as shown in the following sequence:
Weight-loss (g) NaOH solution > NaCl solution
6- The micro hardness of pure (Al) take the following sequence:
Micro hardness (Hv) NaCl solution > NaOH solution
7- The microscopic inspection show different microstructures of the Al metal contributed to the different corrosion behavior in the two corrosive solutions.

Table (1): The results of ultrasonic inspection for Al sample after immersion in 0.1N NaOH.

<table>
<thead>
<tr>
<th>Time (day)</th>
<th>Thickness(mm)</th>
<th>Different in thickness(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (before immersion)</td>
<td>1.92</td>
<td>---</td>
</tr>
<tr>
<td>10</td>
<td>1.20</td>
<td>0.72</td>
</tr>
<tr>
<td>20</td>
<td>0.80</td>
<td>1.12</td>
</tr>
</tbody>
</table>

Table (2): The results of ultrasonic inspection for Al sample after immersion in 0.1N NaCl.

<table>
<thead>
<tr>
<th>Time (day)</th>
<th>Thickness(mm)</th>
<th>Different in thickness(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (before immersion)</td>
<td>2.00</td>
<td>---</td>
</tr>
<tr>
<td>10</td>
<td>1.60</td>
<td>0.4</td>
</tr>
<tr>
<td>20</td>
<td>1.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

Table (3): Values of corrosion potential and corrosion current density and rate of corrosion for pure Al in NaOH and NaCl solution.

<table>
<thead>
<tr>
<th>Medium</th>
<th>(-E_{\text{corr}}) (mV)</th>
<th>(i_{\text{corr}}) (µA/cm²)</th>
<th>(R_{\text{mpy}}) mil/year</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1M NaOH</td>
<td>1620.1</td>
<td>188.19</td>
<td>81.5427</td>
</tr>
<tr>
<td>0.1M NaCl</td>
<td>642.1</td>
<td>23.49</td>
<td>10.1782</td>
</tr>
</tbody>
</table>

Table (4): Results of weight-loss measurement for pure Al samples in the experimental solutions.

<table>
<thead>
<tr>
<th>Medium</th>
<th>Time(day)</th>
<th>Weight(g)</th>
<th>Different in weight(g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1M NaOH</td>
<td>0</td>
<td>0.43528</td>
<td>---</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.32410</td>
<td>0.11118</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.32192</td>
<td>0.11336</td>
</tr>
<tr>
<td>0.1M NaCl</td>
<td>0</td>
<td>0.43248</td>
<td>---</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.42279</td>
<td>0.00569</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.3282</td>
<td>0.10028</td>
</tr>
</tbody>
</table>

Table (5): Results of micro hardness measurement for pure Al samples in 0.1N NaCl solution.

<table>
<thead>
<tr>
<th>Before immersion</th>
<th>After immersion for 10 days</th>
<th>After immersion for 20 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>L Hv</td>
<td>164</td>
<td>160</td>
</tr>
<tr>
<td>4.9N Hv</td>
<td>152</td>
<td>144</td>
</tr>
<tr>
<td>9.8N Hv</td>
<td>135</td>
<td>132</td>
</tr>
</tbody>
</table>

Table (6): Results of micro hardness measurement for pure Al samples in 0.1N NaOH solution.

<table>
<thead>
<tr>
<th>Before immersion</th>
<th>After immersion for 10 days</th>
<th>After immersion for 20 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>L Hv</td>
<td>164</td>
<td>69</td>
</tr>
<tr>
<td>4.9N Hv</td>
<td>152</td>
<td>45</td>
</tr>
<tr>
<td>9.8N Hv</td>
<td>135</td>
<td>Over range</td>
</tr>
</tbody>
</table>
Fig (1): The variation of thickness with time for pure Al in 0.1N NaOH and 0.1N NaCl.

Fig (2): Polarization behavior of pure Al in 0.1N NaOH solution.

Fig (3): Polarization behavior of pure Al in 0.1N NaCl solution.

Fig (4): The variation of weight with time for immersion of pure Al sample in 0.1N NaCl and 0.1N NaOH.

Fig (5): Effect of 0.1N NaCl solution on the Vickers hardness of pure Al.

Fig (6): Effect of 0.1NaOH solution on the Vickers hardness of pure Al.

Fig (7): Microscopic inspection of pure Al before immersion in the corrosive medium 0.1N NaOH solution.
References:
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بعض الاختبارات غير المتلفة لمعدن الألومنيوم في محلول 0.1 عياري كلوريد الصوديوم و هيدروكسيد الصوديوم

سلامة إسماعيل إبراهيم

الخلاصة:
في هذا البحث تم دراسة السلوك التآكلي لمعدن الألومنيوم باستعمال الاختبار غير المتلف والذي هو تقنيه غير متميزة لتحديد كمال المادة. استخدمت في هذا البحث تقنية الموجات فوق الصوتية لقياس التآكل الذي يحدث في الأوساط الاكلية المختلفة (0.1 عياري كلوريد الصوديوم،0.1 عياري هيدروكسيد الصوديوم). كما تم دراسة التآكل بطريقة الحذق بالوزن والطريقة الكهروكيميائية واختبار الصالبة المجهرية لمعدن الألومنيوم بالإضافة إلى الفحص المجهرى للعينات قبل وبعد العصر في الوضع الأكل. كما تم تفسير التغيرات التآكلية لهذه الأوساط والتي تتضمن جهد التآكل وكثافة تيار التآكل. تشير النتائج الي أن محلول هيدروكسيد الصوديوم 0.1 عياري كان عامل تآكل أكثر من محلول كلوريد الصوديوم 0.1 عياري. وتشير فحوصات الصالبة إلى أن الألومنيوم المغمر في 0.1 عياري هيدروكسيد الصوديوم تقل صلادته بمجرد الزخم أكثر مما للألومنيوم المغمر في 0.1 عياري كلوريد الصوديوم.