Study of corrosion in aluminium using neutron radiography technique

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Neutron radiography technique has been adopted for detection of corrosion in aluminium by filling artificially made holes on aluminium slab with Al(OH)₃. The contrast between the optical densities of corrosion products and aluminium slab was assessed from the densitometric measurements. Variation of optical density difference with sample thickness has also been studied. The results confirm that approximately 0.039 mm thick corrosion products having diameter 10 mm can easily be detected in 2 cm thick aluminium slab. The linear attenuation coefficient of Al(OH)₃ has been obtained as 0.9447. From the present investigation it is confirmed that film neutron radiography (NR) technique is helpful for investigation of Al(OH)₃ type corrosion products in aluminium.

1 Introduction

The non-destructive detection of corrosion on aluminium surface is an important technical problem. Neutron radiography is complementary to other conventional radiography techniques such as X-ray, gamma-ray radiography and ultrasonic methods. Moreover, NR has some extra advantages, such as it can detect hydrogenous materials very efficiently due to large neutron absorption cross-sections of hydrogen.

Most types of aluminium corrosion formed for aging, frequent changes of environment and moisture contain hydrogen in various chemical forms. NR technique is very sensitive for detection and investigation of nature and status of corrosion in aluminium. Corrosion in aluminium has been studied by various workers.¹ ²

The objective of the present study is to establish the present facility as an instrument to detect corrosion in aluminium and study corrosion in different materials. Aluminium is almost transparent to neutrons but its corrosion products such as Al(OH)₃ have a high neutron attenuation cross-section. In the present work, we have tried to determine the depth of the corrosion products on aluminium slab, in addition to the detection and localization of the corrosion and detection of minimal thickness of corrosion products on the metal.

2 Attenuation of Thermal Neutrons by Aluminium and its Corrosion Products

The corrosion products of aluminium can be considered as the following transformation:

Al (density 2.7 g/cm³)

→ Various Al trihydrate (Al(OH)₃)

Al monohydrate (Al(OH)) and Al-salts.

In practice Al(OH)₃ (hydrargilite or bayerite) constitutes 60-90% of the corrosion products, and the rest are trihydrates, monohydrates (bohemite, diaspor), oxides, various aluminium salts and organic compounds. Such affinity in Al(OH)₃ dictates for the choice of the compound, among all other products, as the target material for identification of aluminium corrosion.

3 Mathematical Formulation
The relative contrast $K_{ip}$ of the image of an Al(OH)$_3$ inclusion embedded in aluminium base metal is given by formula

$$K_{ip} = \frac{(D_p - D_{al})}{D_{al}}$$

where, $D_p$ is the film density of the image of the corrosion product and $D_{al}$ is the film density of the image of uncorroded surrounding aluminium. Minimum thickness of corrosion product can be detected by using NR through measurement of minimal visually detectable relative image contrast $K_{ip}$ (min). For such a case the minimum detectable thickness $X_{ip}$ (min) is given by:

$$X_{ip}(\text{min}) = K_{ip}(\text{min}) / \Sigma_{ip}$$

where, $\Sigma_{ip}$ is the linear attenuation coefficient of corrosion products for thermal neutrons.

4 Experimental Details

4.1 NR facility

In the present investigation, experiments were performed at the existing NR facility of Bangladesh Atomic Energy Commission. A detailed description of the facility has been given elsewhere$^9$. The facility was assessed in terms of various measuring parameters and the quality of radiographs using the facility was found to be of category-I. The relevant essential parameters$^{10}$ for the facility are given in Table 1. The Cd-ratio for the present work was fixed between 8.58 and 14.39 for which a good quality NR radiographs$^{11}$ are assured. The observed value of $\phi_p/\gamma$ ratio (Table 1) shows that the gamma dose is quite low resulting in much smaller fogging in the image detector. So, the resolution and contrast of the neutron radiographs of the present facility are expected to be reasonably good.

4.2 Samples

In the present work, we have performed the quantitative determination of the depth of the corrosion products on aluminium slabs. For this purpose four drills were made in a pure aluminium sample of size $13.0 \text{ cm} \times 8.7 \text{ cm} \times 2.0 \text{ cm}$ of depths $2$ mm, $4$ mm, $6$ mm and $8$ mm. Diameters of the holes were $10$ mm in each case. For the first set of experiments, the holes were filled with Al(OH)$_3$ powder and the ends of those holes were closed with thin aluminium foils. In other words, we had prepared an artificially made corrosion in aluminium of different thickness on pure Al-plate.

For the second set of experiments we cleared the holes and put Al(OH)$_3$ powder to make a layer of Al(OH)$_3$ of $2$ mm in all the holes and closed the holes by Al-disk having diameter of $10$ mm made from Al-sheet. In this way, we had artificially prepared corrosion of $2$ mm thickness on the surface and at depths $2$, $4$ and $6$ mm.

4.3 NR imaging and processing

During loading the film (Agfa Structurix D4, DW type) and the converter foil (250 $\mu$m Gd) in the radiography cassette, the emulsion surface of the film was kept in contact with the converter foil, and then the radiography cassette was closed tightly. For irradiation, the NR camera was placed at the optimum sample position ($140$ cm from reactor biological shielding assembly) for which the best radiographs were obtained. After making such an arrangement irradiation was done for a duration of $18$ min. This optimum exposure time was found out after performing a series of experiments with different exposure times. While doing so, the reactor was operated at a power of $250$ kW. After the irradiation standard post-irradiation procedure for developing the film was adopted.

5 Results and Discussion

In the work two major studies have been carried out: firstly, corrosion depth measurement through application of densitometric analysis method. The results so obtained were then utilized for measurements of linear attenuation coefficient of the corrosion product for thermal neutron. Secondly, minimal detectable thickness of the corrosion product Al(OH)$_3$ was measured.

5.1 Detection of the corroded region

One may detect the corroded region by measuring relative contrasts between the optical densities of corrosion products and aluminium slab. The positive prints of the neutron radiograph of corroded region in the aluminium slab also offer a scope for corrosion studies. The corroded regions are
darker as it allowed less number of neutrons to pass through compared to that of the uncorroded regions.

The relative contrast \( (K_{r\rho}) \) between the optical densities of corrosion products and aluminium slab was obtained from the densitometric measurements. The results so obtained for 2 mm thick corrosion products at different depths are:

\[ K_{r\rho} = \begin{array}{ll} 
0.111 \text{ at the surface}, \\
0.095 \text{ at 2 mm depth}, \\
0.126 \text{ at 4 mm depth}, \\
0.174 \text{ at 6 mm depth}. 
\end{array} \]

The values of \( K_{r\rho} \) for corrosion products of varying thicknesses are: \( K_{r\rho} = 0.130, 2 \text{ mm thick corrosion} \); \( K_{r\rho} = 0.238, 4 \text{ mm thick corrosion} \); \( K_{r\rho} = 0.320, 6 \text{ mm thick corrosion} \); \( K_{r\rho} = 0.325, 8 \text{ mm thick corrosion} \).

Data so obtained for relative contrasts for varying thickness have been plotted in Fig. 1. The increase in contrast with thickness is expected. However, the deviation from linearity needs some explanation. This may be due to various factors e.g., neutron flux available, the position dependent distribution of neutron and gamma components etc. the dependence of contrast on the type of neutron flux was demonstrated in Ref (5). For a thickness of 5 mm, the contrast values were found to deviate from the maximum value around 1.25 showing a linear variation to 1.35 to a variation of neutron flux. The first one happened in case of a low flux reactor and the latter one for a high flux one. With both facilities, the images were made with gadolinium converter screens and Kodak SR films. The shape of the curve deviating from linearity at high thickness shows a property, which is equivalent to the beam hardening observed in the radiography with the X-ray beam having a continuous spectrum.

From the calibration curve, the slope of the linear portion was considered for the determination of the linear attenuation coefficient of \( \text{Al(OH)}_3 \). The estimated result is 0.9447 cm\(^{-1}\) as against the actual value of 2.40 cm\(^{-1}\). The evaluated attenuation coefficient is found to be appreciably lower. This may be mainly due to the position dependent distribution of the neutron and gamma components.

An appreciably lower value of attenuation coefficient was also previously observed by Tamaki et al.\(^{13}\) for the conventional arrangement of NR technique while investigating neutron attenuation coefficients for iron, copper and aluminium. This was mainly due to the influence of the position dependent distribution of the scattered neutrons and gamma ray components. Tamaki et al.\(^{13}\) proposed a

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**Table 1 - Parameters of the NR facility**

<table>
<thead>
<tr>
<th>Position of beam cross-section</th>
<th>Thermal neutron flux ( \phi_n ) (n.cm(^{-2}).sec(^{-1}))</th>
<th>( \gamma ) dose (mR.sec(^{-1}))</th>
<th>( \phi_n/\gamma ) ratio (n.cm(^{-2}).sec(^{-1}))</th>
<th>Cd - ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centre</td>
<td>1.13 x 10(^5)</td>
<td>0.5055</td>
<td>2.23 x 10(^5)</td>
<td>10.51</td>
</tr>
<tr>
<td>9 cm from centre top left</td>
<td>9.45 x 10(^5)</td>
<td>0.5480</td>
<td>1.72 x 10(^5)</td>
<td>9.73</td>
</tr>
<tr>
<td>9 cm from centre top right</td>
<td>8.19 x 10(^5)</td>
<td>0.5570</td>
<td>1.47 x 10(^5)</td>
<td>8.58</td>
</tr>
<tr>
<td>9 cm from centre bottom left</td>
<td>9.78 x 10(^5)</td>
<td>0.4368</td>
<td>2.24 x 10(^5)</td>
<td>8.67</td>
</tr>
<tr>
<td>9 cm from centre right</td>
<td>7.61 x 10(^5)</td>
<td>0.5600</td>
<td>1.36 x 10(^5)</td>
<td>14.39</td>
</tr>
</tbody>
</table>
new quantitative NR technique which is the combination of removing the scattered neutron contaminant using honeycomb collimator and subtracting the gamma-ray contaminant term from the apparent neutron image by the net transmitted neutrons were obtained. The relation between the net optical film density and thickness for various materials was fairly expressed by a single exponential curve in a transmittance range 1.0-0.01. The attenuation coefficients were then in a fairly good agreement with reference values.

5.2 Minimal detectable thickness

In the present work minimal detectable thickness of the corrosion product was found to be 0.039 mm of 10 mm diameter as against 0.04 mm obtained by Rant et al.14. In their work, the authors also made artificial corrosion in aluminium by making holes of different diameters all having the same depth. However, we have made holes all having the same diameter but varying depths. The minimal detection capability measured by them although is a good result, characteristic of the standard of their facility, but our result definitely advocates a better facility.

5.3 Comparison of NR with X-ray technique

As a technique, NR can be compared here with that of X-rays. Rant et al.14 investigated attenuation properties of aluminium and its corrosion products for soft X-rays and found minimal detectability of 0.2 mm. The present result e.g. 0.04 found for thermal neutrons in NR analysis proves that NR can also detect even up to one-fifth of the finer corrosion depths.

6 Conclusion

In the present work, corrosion in aluminium has been detected by using NR technique. As aluminium is almost transparent to neutrons, whereas the corrosion products have high neutron attenuation properties NR technique could fruitfully be utilized for the detection of Al(OH)₃ type corrosion in aluminium. The present study ensures that the existing NR facility of Bangladesh Atomic Energy Commission is suitable for non-destructive testing and quantitative evaluation of corrosion in aluminium and in detecting any faults in aluminium structure. The results obtained in the present work have enough importance, as this will be an important addition for a complete study as suggested by Lee et al.5.

References

2. Dance W F. Neutron radiography non-destructive evaluation of aerospace structures. Practical Application


