Experimental analysis of Exfoliation rate on Aluminum alloy Al7010

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Abstract: Aluminium alloys are increasingly being used in the manufacture of structures of airplanes and automobiles due to their superior properties such as high strength to weight ratio and excellent mechanical properties. AA7010 Al alloy is a 7xxx series alloy that is mainly used in the aircraft industries. Al 7010 is said to have high tensile strength as well as high fatigue strength. It is also stress corrosion resistant. However due to exposure to extreme marine and industrial environments, the metal may be subjected to corrosion such as exfoliation corrosion.

Exfoliation is a special form of intergranular corrosion that proceeds laterally from the sites of initiation along planes parallel to the surface, generally at grain boundaries, forming corrosion products that force metal away from the body of the material, giving rise to a layered appearance. Exfoliation corrosion may cause heavy damage to aircrafts in the long run. In the present work, the test has been conducted on Al7010 samples for the determination of rate and extent of exfoliation corrosion by subjecting them to artificial corrosive conditions that mimic the actual marine and industrial environments. The samples are heat treated to T6 and T7 tempers prior subjecting them to the corrosive environments, as most of the aircraft materials are subjected to T6 and T7 tempers to enhance the properties before being put to commercial use. Samples are tested according to ASTM G34 and rated by comparing them with standard photographs. Also electrical conductivity tests on the samples have been carried out which reveals that the electrical conductivity of the Al 7010 alloy increases upon heat treatment.

1. Introduction

Aluminium is a metal which is known for its low density, corrosion resistance abilities, electrical and thermal conductivity[1]. It also provides the benefit of being easily machined, casted and extruded. Aluminium has good corrosion resistance because of its property called passivation. In passivation, the aluminium develops a thin layer of oxide on its surface when exposed to air which protects the metal surface from further oxidation. In the present work, exfoliation corrosion tests have been conducted on aluminium alloy Al7010. From literature it is evident that 7XXX series is predominant in zinc. Due to its high strength to weight ratio and exceptional mechanical properties developed due to age hardening, Al 7010 alloy is widely used in making structures of aircraft and automobiles[2].

The materials used for aircraft applications are generally heat treated to T6 and T7 tempers to attain a high strength and desired properties before they are put to practical use. T6 heat treatment increases the
strength of the alloy retaining its ductility whereas the T7 heat treatment increases the hardness of the alloy along with strength but deteriorating its ductility. Hence the samples are heat treated to T6 and T7 tempers before subjecting them to exfoliation corrosion testing[3].

Exfoliation corrosion is a type of intergranular corrosion in which the corrosion initiates and proceeds laterally from the sites of initiation along the parallel to the surface and force the metal away from the body of the metal giving rise to a layered appearance. As a result, the corroded specimen has greater volume than the parent specimen[4]. Aluminium alloys are greatly susceptible to such type of corrosion. In some cases, a different kind of corrosion called pitting corrosion can be formed. Pitting corrosion usually consists of small cavities on the surface of the metal. This kind of corrosion is more localized and is generally difficult to detect hence it is considered more dangerous than other types of corrosion. In the current study an attempt has been made to create an artificial corrosive environment and the susceptibility to exfoliation corrosion, as well as rate of corrosion is determined[3].

2. Experimentation:

The extruded block of Al7010 was purchased and checked for chemical composition using optical emission spectroscopy for its conformance with the standard. Upon confirmation, metallographic test was conducted on the samples to determine the directionality of the grain structures, as exfoliation corrosion generally takes place along longitudinal transverse direction (LT)[3, 5], thus enabling to prepare and position the test surface accordingly.

2.1 Heat treatment cycle:

In the current work, specimen were heat treatment to T6 condition by solution heat treating at 480°C and then artificially aging at 120°C for 24 hours. The T7 temper is achieved through two stages of aging process. One at 110°C for 8 hours and another at 175°C for 14 hours[6].

2.2 Corrosion test according to ASTM G34 standards for conducting EXCO test:

The constant immersion exfoliation corrosion (EXCO) test as per ASTM G-34 standards is prescribed for Al 2XXX and 7XXX series alloys. For testing the exfoliation corrosion of the samples, the specimen were subjected to forced corrosion in very corrosive artificial environments for duration of 48 hours. The artificial environment created is such that, it mimics the environmental conditions of coastal or industrial areas. It is equivalent to the specimen being exposed to such environments for a period of six to nine years. The artificial environment is created by preparing a test solution comprising of chemicals which are highly corrosive, is mixed with sufficient amount of reagent water. The exfoliation corrosion test was conducted on the specimen to determine the accelerated exfoliation corrosion and its rate as per ASTM G34 (EXCO Test) a standard procedure. A non-reactive (plastic container) was utilized to hold the chemicals for the test duration. Specimen were prepared to the following size, 50 x 100 x 30 mm which is a minimum according to the ASTM G34 were arranged in the container, in such a way that they were separated from each other and fitted with a lid to inhibit evaporation of chemicals. The specimen were degreased using kerosene and then their initial weights were recorded. The specimen were masked using 3M scotch Pressure sensitive tape maskant where the surfaces were not to be tested. Sufficient quantity of test solution was used to provide a volume-to-metal surface area ratio of 10 to 30 mL/cm². Then the test samples were immersed in a test solution comprising of 4M (Molar) sodium chloride, 0.1 M nitric acid and 0.5 M potassium nitrate solution which is 234 g of NaCl, 6.3 mL of HNO₃ and 50 g of KNO₃, together which was diluted to 1 L to get a solution of approximate pH of 0.4[7]. With the above said proportionality 13L of the solution was prepared. The specimen were immersed in the test solution at 25°C for a period of 48 hours supported by inert racks and supports in the container and the test surface was set to upward position.
in order to prevent any loss of corroded material from the surface of the test samples. Periodic inspections of the samples were done during test period. The susceptibility to exfoliation of the test samples were determined by visual examination and performance rating by comparing the test specimens with the available standard photographs [7-9].

Photographic images were captured using a camera with specifications of 21 mega pixel, resolution of 1920 x 1080 pixel, 1.1micrometer pixel size with an f/2.0 lens having a frame rate of 30fps, for the specimens which were non heat treated (NHT)- non-corroded, then the images of NHT specimen with corrosion were also captured. Then photographic images of the heat treated T6 and T7 samples, both non corroded (NC) and corroded (C) were captured. The corroded samples were compared with that of the standard images and the performance rating of the samples were determined, when the samples were still moist. Later the samples were cleaned by rinsing using hot water and soaking them in concentrated nitric acid and once again their weights were recorded. On the basis of difference in the weights obtained before and after the exposure, the corrosion rates were calculated for each sample using the given formula.

\[ \text{Corrosion Rate} = \frac{K \times W}{A \times T \times D} \]  

Where: \( K = \) a constant, \((1.00 \times 10^4 \times D \text{ g/m}^2\text{h})\)  
\( T = \) time of exposure in hours, \((48 \text{ hrs})\)  
\( A = \) area in \( \text{cm}^2\),  
\( W = \) mass loss in grams, and  
\( D = \) density in \( \text{g/cm}^3\), \((2.82 \text{g/cm}^3)\)

2.3 Electrical conductivity test:

Electrical conductivity tests of the samples were carried out before and after the exposure in the test solution, to analyse the effect of its variation with corrosion. The electrical conductivity was measured using eddy current instrument (Conductivity meter). The temperature of the test probe, the standard specimen and the test samples were maintained within ± 3°C between each other. The probe is pressed against the surface of the material and the conductivity in %age IACS (International Annealed Copper Standards) was noted by making a comparison between the obtained value and the standard calibrated value using the relation[10],

\[ \sigma = \frac{1}{\rho}, \text{ where } \sigma = \text{conductivity and } \rho = \text{resistivity.} \]  

3. Results and Discussion

3.1 Chemical composition of the Aluminum alloy:

The chemical composition of the aluminum alloy was analyzed using an optical emission spectrometer. The following table shows the chemical composition of the aluminium alloy.

| Elements | Cu    | Mg    | Si    | Fe    | Mg    | Cr    | Zn    | Ni    | Ti    | Zr    | Al   |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|------|
| Actual   | 1.789 | 0.045 | 0.061 | 0.072 | 2.118 | 0.091 | 5.981 | 0.018 | 0.016 | 0.124 | Rema |
| content  | in Wt %|       |       |       |       |       |       |       |       |       |      |
| Specification limits[6] | 1.5- | <0.10 | <0.12 | <0.15 | 2.10- | <0.05 | 5.70- | <0.05 | <0.06 | 0.10- | Rema |
| limits    | 2.0   | 2.60  | 6.70  | 0.16  |       |       |       |       |       |       |      |

The above chemical analysis report confirmed that the chemical composition conforms to the chemical composition of aluminium alloy Al7010.
3.2 Grain orientation:

The aluminum alloy was tested for its grain orientation. This test was carried out in order to cut the specimen in a cutting direction parallel to the grain orientation. The reason being that the exfoliation of the surface of the specimen upon exfoliation corrosion exfoliates parallel to the grain orientation i.e. along longitudinal transverse (LT) direction[5, 11].

![Figure 1. Directionality of the grain structures before etching.](image1)

![Figure 2. Directionality of the grain structures after etching (Keller’s reagent etched).](image2)

3.3 Visual inspection:

After the Al 7010 samples were subjected to corrosion, the samples underwent visual inspection to estimate the extent of corrosion damage. The following codes and classification of rating as shown in the table 2 and table 3 respectively were adhered to when reporting the visual rating of the corroded samples.

Table 2. Visual rating guide for the samples[7]

| Classification       | Codes  |
|----------------------|--------|
| No appreciable attack| N      |
| Pitting              | P      |
| Exfoliation          | EA,EB,EC,ED |

Table 3. Classification of each rating[7]

| Code | Description                                                                 |
|------|-----------------------------------------------------------------------------|
| N    | No appreciable attack                                                        |
| P    | Give appearance of incipient exfoliation.                                   |
| EA   | (Superficial): Tiny Blisters, Thin Slivers, Flakes or Powder, with only Slight Separation of Metal. |
| EB   | (Moderate): Notable Layering and Penetration into the Metal.                |
| EC   | Penetration to a Considerable Depth into the Metal.                         |
| ED   | (Very Severe): Similar to EC Except for Much Greater Penetration and Loss of Metal. |

The following figure 3, figure 4, and figure 5 shows the pictorial images of NHT samples, T6 treated, T7 treated samples after subjected to corrosion. For each type of temper, 4 samples were subjected to
exposure in the corrosive test environment and similar photographic images have been recorded for each type. Only one of the 4 samples images are presented here.

**Figure 3.** Represent samples before corrosion.

**Figure 4.** The non heat treated samples represent pitting (P) type corrosion.

**Figure 5.** The T6 heat treated samples represent (EA) type exfoliation corrosion.

**Figure 6.** The T7 heat treated samples represent (EC) type exfoliation corrosion.
3.4 **Corrosion Rate:**

The corrosion rate for each sample was calculated in order to estimate the extent of corrosion. The initial total surface area and the loss in mass after corrosion were determined. The corrosion rate was calculated using the relation,

\[
\text{Corrosion rate} = \frac{K \cdot W}{A \cdot T \cdot t} \text{ g m}^{-2} \text{ h}^{-1} \quad [7, 8]
\]  

The following table shows the average corrosion rate of each tempered sample. Each reading is an average of four individual samples with three readings per sample of each category.

| Temper | Corrosion rate (g m\(^{-2}\) h\(^{-1}\)) |
|--------|-------------------------------------------|
| NHT    | 7954.875                                  |
| T6     | 19975.54                                  |
| T7     | 28556.55                                  |

The results obtained after the calculation of corrosion rate reveal that, T7 tempered samples have the highest corrosion rate followed by T6 tempered, and then followed by the non-heat treated samples. The increase in corrosion rate of T6 over NHT is 151.11%, the increase in corrosion rate of T7 over T6 is 42.95% and the increase in corrosion rate of T7 over NHT is 258.98%, which indicates that higher the temper of heat treatment, higher the corrosion rate.

![Comparison of corrosion rates](image)

*Figure 7. The graph representing comparison in corrosion rate of different tempered samples.*

3.5 **Effect on conductivity:**

Electrical conductivity test was conducted on the samples before and after corrosion to check for variations in electrical conductivity (of the surface) after the samples underwent exfoliation corrosion. The following graph shows the electrical conductivities of samples before and after corrosion.
Analysis of the graphs reveals that the electrical conductivity increases due to corrosion, as well as the conductivity enhances with the heat treated samples. Heat treatment to the higher temper further enhances the conductivity. Also, the conductivity increases relatively with respect to corrosion and its tempers.

The non-heat treated sample showed a 25% increase in electrical conductivity after corrosion, whereas, the T6 treated samples enhanced the electrical conductivity by 19.01%. The T7 treated samples exhibited a 53.06% increase in electrical conductivity.

The T7 treated samples have the highest electrical conductivity. The reason for this increase in conductivity is due to exfoliation corrosion of the surface, which results in presence of oxidized aluminium ions which act as channels for electrical conduction. The increase in the extent of corrosion results in formation of more oxidized aluminium ions resulting in increased electrical conductivity[10, 12-14].

4. Conclusion

1. Upon visual rating of the corroded specimens it is concluded that:
   - T7 tempered alloy samples show (EC) type exfoliation corrosion
   - T6 tempered alloy samples show (EA) type exfoliation corrosion.
   - Non heat treated alloy samples show pitting type (P) corrosion.

2. Corrosion rate of T7 conditioned samples is higher than T6 conditioned and non heat treated samples. The increase in corrosion rate of T6 over NHT is 151.11%, the increase in corrosion rate of T7 over T6 is 42.95% and the increase in corrosion rate of T7 over NHT is 258.98%.

3. Electrical conductivity of aluminium 7010 samples increased after corrosion by 25% for NHT, 19.01% for T6 and 53.06% for T7 tempered samples.

4. The result supports the designing of various parts of an aircraft, considering the susceptibility to the rate of corrosion, by making use of appropriate tempers.
5. Scope of the work

1. The corrosion tests can be extended with stress as a parameter, i.e. SCC rating can be evaluated.
2. The tempers and the corrosion rates can be optimized, considering other properties of the material.

6. References

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