Ex Situ Tribological and Electro-chemical analysis of Aluminium

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Abstract. Aluminium sample coupon is evaluated for electro-chemical, tribological and microstructural study under selected test conditions. Aluminium is a light-weight material chiefly preferred in fields like automobile, aerospace, marine and satellite domestic appliances etc. Moreover, due to its specific characteristics, it plays a crucial role in industries and research fields. In the present work, the ex situ tribological (wear test), electrochemical (corrosion test), mechanical (microhardness test) and microstructural (nodularity percentage) behaviours of Aluminium sample is presaged. The test results reveal that, when experiment advance, with respect to time the wear rate increases, frictional force is decreased and pin temperature increased. The corrosion test is held on specimen coupon in two modes, before and after wear test, and observed that the corrosion rate gets improved on specimen after wear test. Nodularity analysis also express that the nodularity percentage is increased by 5%. Before wear test, the percentage of accepted count to total count of nodules on texture is 65.18%, while the percentage after wear test is found to be 70.29%. The Vickers Microhardness analysis exhibits the hardness value as 160 HV on Vickers Hardness Scale.

Keywords: Aluminium; Corrosion Rate; Tafel Plot; Nodularity Analysis; Wear Rate.

1. Introduction

Pure Aluminium contains a maximum of 99.9% Aluminium, but it is soft and not preferred for commercial purposes. A tiny percentage of Copper (Cu) is added at a maximum of 0.5% in weight to attain the desired mechanical strength. The Aluminum material is preferred chiefly material to make components in various fields like aerospace, automobile, domestic purpose, marine and many more applications due to its high resistance to corrosion, light-weight etc. [1]

Aluminium is evaluated for its tribological, electrochemical behaviour and microstructural analysis under the influence of different electrolyte media. In general, Aluminium is predicted by exposure to saline solutions. Sodium Chloride-NaCl, Sodium Hydroxide- NaOH, Sodium Fluoride - NaF, with diverse percentage compositions, are preferred as electrolytes. The pH value of the solution, temperature conditions, the surface roughness of the sample are some of the influencing factors of corrosion of Aluminium metal. 1M NaCl, 2% NaCl, 3% NaCl, 4 M NaOH are the concentrations of various solutions used to presage the electrochemical behaviour of the material [2-5].
Researchers are reviewed about various concepts of works held on Aluminium upto now related to type of material, composition of material, various alloying elements added, various electrolytes and working conditions for specific applications. Tribocorrosion is the platform where the material undergoes both tribological and electrochemical behaviour under test conditions. Due to economic characteristics of Aluminium, the industrialists and researchers exhibit more interest on the combined effect of wear and corrosion study. The ASTM G119 standards reveal about the methodology of work to be followed to evaluate tribocorrosion on passive materials, which is followed by many people until 2016. Later the standards are altered by UNE112086 to overcome the drawbacks which are mentioned by many authors regarding the test procedure [6-9].

To reduce the formation of oxide layer on aluminium when the material is exposed to saline environment, the pure aluminium is alloyed with other elements to make it as a composite. Some of the alloying element are Zinc (Zn), Silicon (Sn), Manganese (Mg), Copper (Cu), Iron (Fe) etc to enhance the achieve the desired properties. The series of Aluminium alloys like Al6061, 5A05, AA7075 are some of the Aluminium alloys used as composite materials and evaluated for their tribological and mechanical properties according to the applications [9-12].

The inhibitors play a crucial role in a corrosion test environment. These inhibitors are anodic, cathodic and organic types which it slows down by forming a protecting layer and controls the release of ions to the media in the corrosion process. The anodic inhibitor like tannins and its extracted acid are used as inhibitors and presaged the corrosion rate of material under acidic media. The derivates of pyridine also tested as an inhibitor with various environments like NaCl, HCl and NaOH [13,14].

Advanced manufacturing processes reduce the man power usage, number of tools used, reduces time, post processing work and wastage of raw materials. Additive Manufacturing (AM) is one of the advanced manufacturing processes which is quiet opposite to that of conventional manufacturing like forming and subtractive machining. Some of the materials which are suitable for additive manufacturing process are titanium and its alloys, Cobalt-Chromium alloys, Inconel and its alloys, Aluminium and its alloys, Copper and its alloys etc. Beyond these, ceramics, polymers, are also preferred in AM process. Laser Engineered Net Shaping (LENS) is one of the advanced AM process suitable to fabricate the desired component with near net shape and full density. Laser power is used to melt and deposit the raw material which is either in powder or wire form on substrate plate. This process happens in a controlled atmosphere. Co-Cr-W and Co-Cr-Mo alloys are deposited on stainless steel substrate with desired shape and the samples are tested for tribological, mechanical, electrochemical and optical measurement [15,16].

Stir Casting Process (SCP) is one of simple and modern manufacturing process used to fabricate Metal Matrix Composites (MMCs). The light-weight materials like magnesium and aluminium are mostly preferred with this process at a working temperature range from 800-950°C. In this process the alloying elements are added as addons when the parent material is in molten state. The stirring process is done to achieve a homogeneous mixture of composite as outcome. Magnesium alloys AZ31, AZ91, AZ90, and aluminium alloys like AI7075, Al-TiC are some of the light weight alloys usually preferred to make MMCs using this process. Titanium Carbide, Al₂O₃, SiC, Zn are some of the add-ons preferred to add as alloying elements [17,18]. The electrochemical behaviour of other ferrous and non-ferrous materials is also predicted under the influence of sodium chloride solution [19,20]. The wear rate of material always depends of chemical composition of two sliding materials. This wear occurs due to friction generated between materials contact surface and softer material surface will get worn-out due to friction generation. Pin on Disc (PoD) apparatus is used to predict the wear rate, frictional force and coefficient of Friction (CoF) of different materials by altering the test parameters like speed of disc in revolutions per minute (rpm), applied load in newtons, distance travelled by pin, and so on according to the applications [21,22].
2. Materials and Methods
2.1 Specimen Material:
The Aluminium material collected from the commercial market is machined to shape and size shown in Fig-1. The chemical composition of Aluminium is shown in Table-1. After machining, the specimen is prepared to a cylindrical shape for wear test, a flat piece for corrosion test, microhardness test, and microstructural analysis. The specimen surface is ground for removing macro and micro level particles. The texture is cleaned with acetone solution to degrease it. The specimen sizes are 40mm length X 12mm diameter for wear test, circular disc shape for corrosion test with 5mm thick and 12mm shown in Fig-1.

In the present work, the specimen coupon is tested for polarisation technique like Tafel Plot, hardness test like Vickers Microhardness test. Moreover, microstructural analysis like the Nodularity percentage test and tribological test like wear test is conducted using proper suitable apparatus and ASTM standards.

Table-1. Chemical Composition and Properties of Aluminium (in % wt.)

| Material                  | Pure Aluminium | Copper | Mechanical Strength | Young’s Modulus |
|---------------------------|----------------|--------|---------------------|-----------------|
| Min. %                    | 99.5           | 0.12   | 7                   |                 |
| Max. %                    | 99.9           | 0.5    | 11                  |                 |

Table 2: Terminal connections between corrosion cell with Potentiostat

| SL. No | Terminal Colour | Electrode Description       | Experimental connection       |
|--------|-----------------|------------------------------|-----------------------------|
| 1.     | Red Colour      | Working Electrode (WE)       | Specimen Coupon             |
| 2.     | White Colour    | Counter Electrode (CE)       | Ag/AgCl₂ (Silver: Silver Chloride) |
| 3.     | Blue Colour     | Reference Electrode (RE)     | Platinum Electrode           |

2.2 Polarisation Curve:
Tafel plot is the curve drawn between the logarithmic scale of current on abscissa to corrosion potential on the ordinate. The curve undergoes two processes, oxidation and reduction, with Anodic and cathodic regions. Open Circuit Potential (OCP) is the primary voltage where no electricity supply is given to the circuit.

In the present work, the Aluminium specimen is cleaned with acetone solution and placed in an electro-chemical corrosion cell made of glass. Three electrodes (Working Electrode, WE; Counter Electrode, CE and Reference Electrode, RE) are connected with corrosion cell attached with Potentiostat Apparatus (Make: Biologic SAS, France; model: SP-300). The terminal connections with electrodes are summarized in Table-2. The Working Electrode is connected to Aluminium Specimen. Platinum meshed Electrode is used as Reference Electrode, and Ag/AgCl₂ is used as Saturated Calomel Electrode (SCE), which acts as a Counter Electrode (CE). 3.5% Sodium Chloride Solution (NaCl) is filled in the glass electro-chemical cell. The terminal electrode connections are shown in Fig-2.
2.3 Wear Test
Pin on Disc (PoD) is apparatus used to predict the wear rate of material. In the current experimentation, PoD (Make: DUCOM, Bangalore) is used to presage the wear rate of the Aluminium sample. The pin is stable in this apparatus, and the disc will rotate against the pin with the desired speed. The disc is made of EN31 Hardened Steel with a Roughness Value of 1.6Ra and a surface hardness value of 62HRc. The pin is primarily cleaned with acetone solution to remove dust particles after machining to size. The pin shown in Fig-1 is 12mm diameter and 40mm length fixed in 12mm pin holder, and the pin holder is assembled with apparatus. The selected test parameters are labelled in Table-3. The disc speed, distance travelled by pin and track diameter at which the pin is placed on the disc are the three parameters that are assumed before experimentation.

At present, the disc speed is taken as 500 rpm, the track diameter as 70 mm, and the distance travelled by the pin is 1.5 km. with these assumed values, the time to conduct the wear test is calculated, and the obtained time value is mentioned in Table-3. An external load of 30N is applied to the pin to experiment. The experimental setup PoD is shown in Fig-3 attached with the EC unit.

| Sl. No | Pin Material | Track Diameter | Disc Speed | Distance Travelled by Pin | Test Run Time (apprx.) |
|--------|--------------|----------------|------------|--------------------------|------------------------|
| 1.     | Aluminium    | 50mm           | 500rpm     | 1km                      | 14min                  |

2.4 Hardness Test:
Hardness is the resistance offered by material against externally applied load. It depends on the chemical composition of materials. Additives are used as add-ons to improve the mechanical, tribological properties of soft materials. Types of hardness tests conducted on materials are Brinnell, Rockwell,
Vickers and Knoop etc. But the microhardness is performed on the specimen with meagre load conditions. Vickers Microhardness test is used to achieve the hardness test. An external load of 300gms is applied on the surface of the sample for 10 seconds.

2.5 Microstructural Analysis:
The Metallurgical Microscope (Make-METASCOPE, Bangalore) is used to check the microstructure of the Aluminum specimen surface at 100X. The texture is matched before and after the wear test is conducted using PoD on the specimen. Nodularity percentage is checked on the surface, before and after the wear test using DWinter Material Plus Software (Ver: 4.5), and the results are plotted.

3. Results and Discussions:
3.1 Comparative Tafel Plot Curve (before and After wear test)

![Comparative Tafel Plot Curve of Aluminum](image)

**Table 4. Tafel Plot Slope Values and Corrosion Rate**

| Parameter | $E_{corr}$ | $I_{corr}$ | beta c | beta a | Corrosion rate | Corrosion rate |
|-----------|------------|------------|--------|--------|----------------|----------------|
| Units     | mV         | $\mu$A     | mV     | mV     | mmpy          | mpy            |
| Value     | -761.341   | 0.655      | 108.1  | 45.4   | 9.513e-3       | 0.374 478      |
3.2 Wear Test

![Fig-7 Time Vs Wear rate](image)

![Fig-8 Time Vs Frictional Force](image)

![Fig-9 Time Vs Pin Temperature](image)

![Fig-10. Comparative Graph](image)

3.3 Vickers Hardness:

| Specimen   | D₁  | D₂  | HV  | HRC | HRB |
|------------|-----|-----|-----|-----|-----|
| Aluminium  | 58.95 | 58.95 | 160 | 3   | 84  |
3.4 Microstructural Analysis:

3.4.1 Nodularity Test (Before and After Wear test)

![Fig-11. Surface texture at 100X (before Wear test)](image1)

![Fig-12. Nodularity Analysis (Before Wear test)](image2)

![Fig-13 Surface Texture at 100X (After Wear test)](image3)

![Fig-14 Nodularity Analysis (After Wear test)](image4)

3.5 Discussions

- Before wear test - The corrosion rate shows as 9.51314 e-3 in millimetre per year (mmpy) and Corrosion Potential (E_{corr}) and Corrosion Current (I_{corr}) values are -761.341 Volts and 0.655 µA
- After wear test - The corrosion rate shows as 7.30985 mmpy and Corrosion Potential (E_{corr}) and Corrosion Current (I_{corr}) values are -742.743 mV and 503.299 µA
- Here, two separate samples are tested for polarisation technique (Tafel Plot), one sample is tested for corrosion test (i.e., sample before wear test) and other sample is tested for Tafel plot where the sample is tested for wear test (i.e., sample after wear test)
- From Fig-6, for the specimen (before wear test) the Open Circuit Potential (OCP) line occurred at -0.77 Volts for Tafel plot (blue colour) curve.
- Similarly, the Open Circuit Potential (OCP) line occurred at -0.74 Volts point on Y-axis on sample tested after wear test. The difference in OCP point is observed from Fig-6 which is due to the release of more ions from material surface due to surface disturbance after wear test to the electrolyte and enhances the process time to get stabilise the process
The wear rate initiated at a minimum of -15 microns and reached to a maximum of +20 microns, pin temperature reached from 35.4 to 36.4 °C and frictional force exhibits downfall from 11N to 9N.

From Fig-13, the parallel inclined strips of lines are ploughing type of structure occurred on the surface of specimen which is a clear indication of occurrence of abrasive wear.

Microhardness value is obtained as 160HV.

From Fig-14, after the wear test on sample, it is observed that the Nodularity percentage gets increased from 65.18% to 70.29%, due to abrasive wear the wear debris is formed on that surface which increased the nodularity percentage.

4. Conclusions

Corrosion rate increases from $9.5131 \times 10^{-3}$ mmpy to 7.3095 mmpy for the tested specimen after wear test exhibited as due to surface roughness.

Wear rate has found to be 20 microns maximum due to applied load, and due to frictional force generated between surfaces and also pin temperature has reached a maximum of 36.4°C.

Micro hardness Value reaches as 160HV implies that the material is so smooth and less resistance to applied force.

Nodularity percentage is increased (65.18 to 70.29%) from when the sample is tested after wear test due to presence of impurities in the material and formation of wear debris formed during sliding motion of steel disc and got stuck to the surface of sample.

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