Evaluation of Fracture Toughness and Mechanical Properties of Aluminum Alloy 7075, T6 with Nickel Coating

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Abstract

A study on mechanical and fracture properties, through the experimentation techniques has been carried out on a Al 7075-T6 and it coated with an Electroless Nickel deposits of 10 – 20 μm in coating thickness. In the recent years there has been considerable interest in the Electroless Nickel plating due to its extensive use in electronics, automobiles, aerospace and other industries. Electroless Nickel plating is a controlled autocatalytic reduction of nickel ions using suitable reducing agent such as sodium hypophosphite. ASTM Standard – E 399 for plane strain fracture toughness determination was adhered in this investigation. The results obtained, show that the EN coating can give rise to a significant improvement in the fracture behavior (performance) of the substrate and also it has shown that EN coating exhibit a very good adhesive property to the alloy material when the tensile stresses exceeding the yield strength are applied to the system and performance of these pretreatments was studied by SEM analysis.

Keywords: Electroless Nickel coating; 7075-T6 aluminum alloy; Fracture behavior; SEM analysis;

1. Introduction

Aluminum and its alloys have found widespread applications in automobile and aerospace industries, because of their excellent properties such as high strength to weight ratios, high thermal conductivity and good corrosion resistance. High strength aluminum alloys, such as 7075-T6, are widely used in aircraft structures due to
their high strength-to-weight ratio, machinability and low cost. These are widely used for high strength structural applications such as aircraft wing skins and internal supporting members as well as missile components and automobile industries [1-7]. However, due to their compositions, these alloys are susceptible to corrosion. Corrosion is a major concern involving the structural integrity of aircraft structures. Corrosion has been shown to reduce the life expectancy of these structures considerable. Aircraft, during normal operation, are subjected to natural corrosive environments due to temperature, humidity, rain, and seawater.

DeBartolo and Hillberry [8] established that in many aluminum alloys used in the aircraft industry, fatigue cracks could be nucleated not only at macroscopic defects, such as machining marks and corrosion damage, but also at crack constituent particles inherent to the alloy, formed during the solidification process.

In recent investigation, K Brunelli et all [9] made a research on Ni coated and subsequent diffusion heat treatments studies for Al 7075 with Ni coatings of different thickness on electrolytic and electroless process. EN coated Aluminium was investigated for its tribological properties through experimental methods and strength through Finite Element Analysis [10]. Fatigue behavior of a 7075-T6 aluminum alloy coated with an electroless Ni–P (EN) deposit, in the as-plated condition, of approximately 38–40 μm in thickness and a high P content, of approximately 18 wt%. The results obtained, show that the EN coating can give rise to a significant improvement in the fatigue and corrosion–fatigue performance of the substrate, depending on the testing conditions.

The investigation shows that EN coated Aluminium has superior tribological properties when compared with Cast Iron and it has adequate strength. It is concluded that EN coated Aluminium has a high potential to be used as cylinder liners. Investigation on Dry sliding wear behavior of electroless nickel-phosphorus (EN) coating of thickness 35 μm deposited on 7075-T6 aluminum alloy. The surface morphology of the pretreatments was studied by a confocal laser scanning microscope. The performances of these pretreatments of EN were evaluated by dry sliding wear studies and followed by SEM studies. Ni strike provided better interlocking adhesion between EN and Al and this pre-treatment noticeably improved the wear, frictional and hardness behavior of the EN coatings on 7075 Al substrate and further enhanced it by heat treatment of 400 °C/h. Prasanth sahu et all carried out a research on tribological electroless nickel coatings on alloys. The advantages include uniform coating and also nonconductive materials can be coated. High hardness, good wear resistance and corrosion resistance make EN coated materials for tribological applications in aerospace, marine, automobile, electrical and chemical industries [11-19].

According to ASTM E-8M and ASTM E-399 standards, experimentally the influence of EN coating on fracture and mechanical behavior of Aluminum 7075-T6 alloy was investigated [20]. Specimens were coated with 10 and 20 μm of EN. Both coated and uncoated specimens were tested on a UTM, load-crack opening displacement and load–elongation and values were recorded. According to the ASTM E-399 standard testing procedure the nickel coated CT specimens of different thickness are tested in plane strain condition under Mode 1 failure analysis in TL orientation, the fracture toughness is evaluated with the principles of Linear Elastic Fracture Mechanics (LEFM) and also evaluate the mechanical hardness properties. For a valid value of plane strain fracture toughness, the amount of shear lip fracture must be negligible in comparison with the total thickness of the specimen. The measured fracture toughness \( K_Q \) should satisfy the following condition according to the test standard validity criteria: (a) the specimen geometry validity, i.e. \( B \geq 2.5 \ (K_Q/\sigma_U)^2 \), and (b) the load – displacement curve validity in which \( P_{max} P_Q \leq 1.10 \), where \( B \) is the specimen thickness, \( P_{max} \) is the maximum load sustained by the test specimen and \( P_Q \) is the load at 5% secant offset. Fracture and mechanical behaviour of a 7075-T6 aluminum alloy and to compare the coated system with that of the uncoated substrate. Analysis of fractured surface using scanning electron microscope in order to understand the nature of failure.

### Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>Al</td>
<td>Aluminum Alloy</td>
</tr>
<tr>
<td>TL</td>
<td>Transverse and Logitudinal</td>
</tr>
<tr>
<td>EN</td>
<td>Electroless Nickel</td>
</tr>
<tr>
<td>CT</td>
<td>Compact Tension</td>
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<tr>
<td>LEFM</td>
<td>Linear Elastic Fracture Mechanics</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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<tr>
<td>KQ</td>
<td>Mode I Fracture Toughness (MPa√m)</td>
</tr>
<tr>
<td>B</td>
<td>Thickness of the Specimen (mm)</td>
</tr>
<tr>
<td>PQ</td>
<td>Critical Load (kN)</td>
</tr>
<tr>
<td>σU</td>
<td>Ultimate Tensile Strength (MPa)</td>
</tr>
<tr>
<td>σY</td>
<td>Yield Strength (MPa)</td>
</tr>
<tr>
<td>Pmax</td>
<td>Maximum Load (kN)</td>
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</table>
2. Experiment Details

2.1. Material Preparation and Electroless Nickel Coating Process

Cast Al 7075-T6, supplied as a block was used in the present study. The typical chemical composition was as follows: 5.9 wt. % Zn, 2.8 wt%. Mg, 0.19 wt. % Fe, 0.09 wt. % Si, 0.02 wt. % Ti, 1.9 wt. % Cu, 0.03 wt. % Mn, 0.2 wt. % Cr and the balance aluminum.

Electroless (autocatalytic) plating consists of a chemical reducing agent in solution to reduce metallic ions to the metal state. Further autocatalytic nickel plating is commonly referred to as Electroless Nickel plating. In contrast with electroplating, Electroless nickel (EN) plating does not require rectifiers, electrical current or anodes. Deposition occurs in an aqueous solution containing metal ions a reducing agent, chelates, complexing agents and stabilizers. Chemical reactions on the surface of the part being plated cause deposition of a nickel alloy. Firstly the surface is subjected to a pretreatment process where the series of chemicals are applied and surface is cleaned to remove oils and other corrosive elements. Sodium hydroxide chemical composition of Electroless Nickel plating chemical composition is shown in Table 1.

The following pretreatments were performed separately on the three samples under the below mentioned conditions:

a) Nickel immersion: A thin film of Ni will be formed on the Aluminium alloy by a reaction between surface of the alloy and nickel salt solution (contact reduction type) after the nickel coating solution is maintained at room temperature for 60 seconds, where the nickel coating contains hydrogen fluoride, boric acid and nickel salts. The Aluminium substrate thus produced effectively on the Ni nucleus acts as a catalyst for the succeeding EN coating.

b) Zinc immersion: Zinc Sulphate, sodium potassium Tartarate and sodium hydroxide is used for preparing Zinc Solution and the process is at ambient temperature for 60 seconds. A thin film of Zinc will be formed on the Aluminium alloy and thus protects the aluminum from re-oxidation. Sodium hydroxide absorbed layer: Sodium hypophosphite and lactic acid at pH 4.5 is used to prepare pretreatment solution and a thin film of hypophosphite will be produced on the aluminum alloy after the process is maintained at a temperature of 80 °C for 5 minutes. Thus formed layer acts as a catalyst for the succeeding EN coating.

At a rate of 5 microns/hour to a maximum of 40 microns/hour, EN coating can be deposited on the aluminum alloy and EN coating has no limitations in terms of thickness due to its continuous process. It is seen that with the increase in the thickness adhesion between the substrate and coating will become significantly weak.

<table>
<thead>
<tr>
<th>Component</th>
<th>Electroless Bath</th>
</tr>
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<tbody>
<tr>
<td>Ni²⁺</td>
<td>5</td>
</tr>
<tr>
<td>Sodium Potassium Tartarate</td>
<td>65</td>
</tr>
<tr>
<td>Sodium Hydroxide</td>
<td>40</td>
</tr>
<tr>
<td>Ammonium Hydroxide (28%)</td>
<td>120 mL⁻¹</td>
</tr>
<tr>
<td>Sodium Borohydrde</td>
<td>0.75</td>
</tr>
<tr>
<td>TINO₂ (mg L⁻¹)</td>
<td>50</td>
</tr>
<tr>
<td>Pb (NO₃)² (mg L⁻¹)</td>
<td>10</td>
</tr>
<tr>
<td>pH</td>
<td>5</td>
</tr>
<tr>
<td>Temperature</td>
<td>80 - 90°C</td>
</tr>
</tbody>
</table>

2.2. Heat Treatment Technique

The zinc is the element that has high degree of solubility with aluminum and thus enhances the fluidity of the alloy [3, 4]. In practice, 5 to 7 % of Zinc is blended with aluminum in order to attain good mechanical properties [3]. Further, small proportions of copper and magnesium resulted in best castability. The temperature for solution heat
treatment for Al 7075 alloys is maintained at a range of 500 to 550°C, because the high temperature results in incipient melting of Zinc rich phase, thereby reduces the mechanical properties. Aluminum with 5.9% Zinc is heat treatable, at eutectic temperature of 500-550°C the alloys containing Zinc will respond to heat treatment by age hardening. The intermediate phase ө is a composition that closely corresponds to Al 7075. Artificial aging treatment (T6) was applied to cast alloys with a sequence of solution heat treating, quenching and artificial aging. Solution heat treatment is performed by heating the alloy into the α-single phase region at a temperature of 500 to 550°C for 8 hours which was further subjected to water quenching in T6 and aging at a temperature of 165°C. Annealing process was carried out up to 170°C for 8 hours and cooled to room temperature. The strength and hardness properties are increased due to precipitation in ө phase.

2.3. Tensile and Hardness

Tensile specimens were prepared from heat treated cylindrical rods, measuring a length of 300 mm and diameter of 20 mm for each of the specimens were prepared in TL orientation. Tensile specimens of circular cross-section with a diameter of 12.7 mm and a gauge length of 30 mm as per ASTM E-8M are machined.

Fig. 1. Dimensions of Tensile test specimen

Fig. 1, shows the dimensions of the tensile test specimen. The tests were performed on a computerized servo-hydraulic universal testing machine. The specimens underwent the tests in displacement control mode, at a rate of 0.1 mm/min. The readings of load against the displacement were measured.

MRB 250 model of Meta test make Rockwell hardness tester was used with a ball indenter of diameter 1/16” and results are measured on the B-scale at 100kgf load according to ASTM E-18.

2.4. Fracture Toughness Test

The test procedure entails the investigation of the plane strain fracture toughness (Klc) of metals by tests using a various fatigue-cracked specimens. The fracture behavior of a material is dependent on the crack orientation and direction of crack propagation relative to the anisotropic property of the material. Pre-cracked notched specimens subjected to fatigue under tension is used as a part of this test procedure Linear Elastic Fracture Mechanics is used to study the elastic deformation through linear stress strain relations for the entire body.

Fracture toughness tests were performed according to ASTM E399 standard testing procedure using CT specimens. Fig. 2, shows the dimensions of the CT specimen. All the specimens were pre cracked from the wire cut notch at 10Hz with load ratio of 0.5 (R). The pre-cracking procedure fulfilled all the restrictions imposed by the standard.
The tests were conducted on ‘BISS’ servo hydraulic static and dynamic testing machines. When the pre-cracked specimen is subjected to external load the crack tip opens due to the tensile stress. Then, plastic deformation blunts resulting in a stretched zone, while voids begin to form ahead of the crack tip. The stretched zone continues to increase and the growth of void will continue until a critical size is reached. The propagation of the crack tip begins by linking of the voids with the blunted crack tip [21]. The ratio of maximum value of $P_{\text{max}}/P_0$ is lesser than 1.05 confirms that the results are valid for $K_{\text{IC}}$. All the calculated values of the fracture toughness are well within the limitations for a valid test shown in table 2.

3. Results and Discussions

3.1. Mechanical Properties

From the Fig. 3, observed that, it is observed that the tensile strength of the EN coated aluminum alloy is higher than that of their uncoated Aluminum alloy. It is also observed that the increase in the thickness of the coating contributes in increasing the tensile strength and the load carrying capacity of the material. Uncoated Al 7075-T6 alloy shows a yield strength of 559 MPa, after EN coating on alloy of 10μm and 20μm yield strength of alloy reaches to the 569 MPa and 603 MPa respectively. It was found that the tensile strength of EN coated alloy increased by 5% than comparing with the uncoated Al 7075-T6. The coating experiences progressive delamination and fracture at the maximum load, due to the tensile stress, resulting in plastic deformation.

From the Fig. 3, it is evident that hardness of the EN coated alloy is greater than that of uncoated aluminum alloy. Further, it can be observed that the 20μm coated alloy exhibits higher hardness compared to that of 10μm coated alloy. It clearly shows that Hardness increases with the increasing thickness of the EN coating.
3.2. Fracture Properties

The coating on the specimen, post fracture appears to be severely cracked and extensively delaminated from the Al7075 alloy surface in various regions, specifically at the crack tip region of the sample. Due to poor ductility of EN coating when compared to the Al7075 alloy surface to deform plastically, the EN coating thus has limited life over the substrate [11]. Fig. 4(a) depicts a basic view of the fracture surface of a specimen in as cast as condition. It is observed that the region of fracture surface was irregularly pulled out due to the tensile load, dominated by a single crack. Fig. 4(b) illustrates a microscopic view of the crack origin, which depicts the EN coating on the Al7075 alloy substrate with uniform thickness 20 μm. It is observed that the nature of the crack deviates from the nature of the pre-crack i.e., unstable crack growth due to the strong adhesion between the EN coating and the Aluminum alloy. It is apparent that such defects were formed during the EN coating process and could be accompanied with the high standard deviation of the number of cycles to failure at constant maximum alternating stress [11].
When the alloy was coated with 10 μm of EN, the fracture toughness was improved when compared with the uncoated aluminum alloys. Further improvement in fracture toughness was observed when coated with 20 μm of EN.

Table 2 shows that uncoated aluminum alloy critical load \( (P_0) \) carrying capacity are 4.4 kN which corresponds to \( K_{IC} \) of 22.8 MPa√m. Further for 10μm & 20μm coated aluminum alloy has a critical load of 6.67 kN and 7.41 kN which corresponds to \( K_{IC} \) values of 34.48 MPa√m & 37.67 MPa√m respectively. It clearly shows that the fracture toughness of the coated alloys increases with the increase in the thickness of the EN coating. This is because of the strong adhesion between coating and the alloy.

<table>
<thead>
<tr>
<th>Material</th>
<th>Coating Thickness (μm)</th>
<th>( P_0 ) (kN)</th>
<th>( P_{max} ) (kN)</th>
<th>( K_{IC} ) (MPa√m)</th>
<th>( K_{IC} ) (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7075-T6</td>
<td>---</td>
<td>4.44</td>
<td>4.73</td>
<td>22.28</td>
<td>22.28</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>6.67</td>
<td>6.92</td>
<td>33.48</td>
<td>33.48</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>7.4</td>
<td>7.74</td>
<td>37.04</td>
<td>37.04</td>
</tr>
</tbody>
</table>

3.3. Fractographic Study

The fracture surfaces are examined under Scanning Electron Microscope. Al 7075-T6 as cast as specimen showed a stable and unstable crack growth region, EN coated (10 and 20 μm) showed only unstable crack growth region. The stable crack growth region is relatively flat, smooth and will not show any deep dimples Fig. 4 (a). The unstable crack growth region is rough, showed deep dimples and pulled regions. This was relatively bright as shown in Fig.4 (b) and Fig.4 (c).
4. Conclusions

The mechanical and fracture properties as investigated for Al 7075-T6 coated alloy shows an exceptional linear relationship between the yield strength, ultimate tensile strength and hardness values. Based on the above results it was found that, with the increase in thickness of the EN coating fracture toughness and tensile properties of Al 7075-T6 alloy increases and it is observed that the nature of the crack deviates from the nature of the pre-crack i.e., unstable crack growth due to the strong adhesion between the EN coating and the Aluminum alloy. Hardness values could be used as an interpreter for tensile strength of this alloy. The scanning electron micrograph of cracked surface of the EN specimen showed relatively higher stable crack growth compare to uncoated alloy.

5. Acknowledgement

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References