Influence of Environment, Residual Stresses on the Fatigue Behavior of 7075-T6 Aluminum Alloy

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**Abstract:** Diamond-like coating (DLC) of 2.5 micron thick coated on the aluminum alloy samples, were subjected to rotating bending fatigue test in methanol and air environments respectively. DLC coating is X-ray amorphous in nature as revealed by grazing incidence X-ray diffraction studies. Residual stresses generated due in the coatings were measured using Raman peak shift analysis of G band of carbon. Fractographic analysis of uncoated fatigue tested samples in methanol revealed severe damage in contrast to coated samples in methanol. Diamond polished and DLC coated samples revealed better coating adherence and fatigue performance than 1200 grit polished and fatigue tested samples.

**Introduction**

Al-Zn-Mg-Cu alloys (7000 series alloys) are widely used in aerospace applications because of its superior mechanical properties achieved through optimized heat treatment methodologies [1]. However, these alloys are susceptible to environmental-assisted degradation when exposed to corrosive environment during static and cyclic loading. To alleviate the environmental influence on material performance, various coating techniques such as PVD-WC/C and DLC coating has been envisaged for improving the performance of the substrate material in ambient and corrosive environments respectively [2].

DLC coating which is a mixture of sp\textsuperscript{2} (graphite-like) and sp\textsuperscript{3} (diamond-like) bonds of carbon, improves hardness and wear resistance over the aluminum substrate for sliding contact applications and corrosion protection [3,4]. The present work focuses on the possible influence of the residual stresses on the fatigue performance of DLC coated Al-Zn-Mg-Cu substrate deposited by physical vapor deposition technique. Also, fractography of the samples provide useful insights into the understanding of the fatigue behavior of the samples.

**Material and Methods**

DLC coated and uncoated samples of aluminum alloy 7075-T6 were subjected to rotating bending fatigue test in air and methanol environment using hourglass specimen geometry. In order to study the influence of substrate surface condition on the adherence of the coating, substrate was polished with either 1200 grit (rough surface) or diamond paste (smooth surface) prior to coating deposition process. Fractographic analysis of fatigue fractured samples were carried out using Quanta 400 scanning electron microscopy (SEM) in backscattered (BSE) mode using solid state detector. Energy dispersive spectroscopy (EDS) was used for elemental and compositional analysis of precipitates and dispersoids in aluminum alloy.
Grazing incidence X-ray diffraction (GIXRD) was carried out in Rigaku Smartlab diffractometer using Cu-Kα radiation and Goebel mirror was used to achieve parallel beam configuration. The voltage and current settings were 40 kV and 100 mA respectively. Grazing incidence angle of $\alpha = 1^\circ$ to $8.5^\circ$ was used to probe the nature of DLC coating. X-ray diffraction spectra was obtained in the range of $15^\circ$- $90^\circ$ ($2\theta$ angle) with an step size of $0.01^\circ$ and scan speed of $10^\circ$/min respectively.

Raman fingerprinting analysis of DLC coated samples was carried out and residual stresses were calculated based on the peak shift of D band and G band of stressed samples with respect to stress-free condition [5]. An argon-ion laser with an excitation wavelength of 488 nm was used for the analysis using a LabRam-HR 800 Raman micro-spectrometer. Signals were collected in the range of 600 cm$^{-1}$ to 2400 cm$^{-1}$ respectively with an integration time of about 2 minutes at 15mW power. Prior to analysis, calibration was carried out using a standard (100) orientated silicon wafer. A Guassian-Lorentzian polynomial function was used for fitting convoluted G and D peaks after background correction using LabSpec (version 5.28) Raman spectroscopy software.

Results and Discussions

Grazing incidence X-ray diffractograms (GIXRD) collected from the DLC coating of 2.5 micron thickness at an incidence angle of $1^\circ$ and $3^\circ$ revealed X-ray amorphous nature of the coating as shown in Fig. 1a. At higher grazing incidence angles, only diffraction peaks corresponding to the aluminum alloys were revealed. Concomitant observations from Raman spectroscopy analysis of the DLC coated samples provides useful insights on the residual stress generated due to coating. Earlier report suggested the presence of internal stresses generated due to coating process affects the mechanical behavior and corrosion behavior of coated substrates [6].

![Fig. 1 Characterization studies on DLC coating (a) GIXRD (b) SEM (c) Raman spectroscopy.](image)

Due to amorphous nature of the coating, D and G peaks of carbon were convoluted as revealed by Raman spectra of as-DLC coated samples as shown in Fig. 1c. The obtained spectral shape is typical of a hydrogenated a-C:H carbon. D and G peaks were deconvoluted to calculate the residual stresses generated due to the coating and it was around $-1.29$ GPa ± 0.1 GPa (compressive).
Fractography of fatigue tested samples showed characteristics fatigue features such as (1) crack initiation from the surface or sub-surface region (2) crack propagation zone and (3) final overload fracture. Irrespective of test environment and surface conditions, all the three distinct zones were clearly delineated in all fatigue tested samples. Final overload fracture was characterized by the presence of dimples. The nucleation sites for each of the dimples were found to be iron-based dispersoids revealed through EDS analysis.

Table 1 Fatigue limits of coated and uncoated samples tested in air and methanol environment.

<table>
<thead>
<tr>
<th>S No</th>
<th>Coated/uncoated</th>
<th>Environment</th>
<th>Surface condition</th>
<th>Stress [MPa]</th>
<th>Number of cycles to failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Uncoated</td>
<td>methanol</td>
<td>1200</td>
<td>230</td>
<td>1702378</td>
</tr>
<tr>
<td>2.</td>
<td>coated</td>
<td>methanol</td>
<td>1200</td>
<td>230</td>
<td>500640</td>
</tr>
<tr>
<td>3.</td>
<td>coated</td>
<td>air</td>
<td>1200</td>
<td>240</td>
<td>6973394</td>
</tr>
<tr>
<td>4.</td>
<td>coated</td>
<td>air</td>
<td>Diamond polished</td>
<td>240</td>
<td>9131116</td>
</tr>
</tbody>
</table>

A magnified view of the crack initiation site of all samples (coated and uncoated samples in air and methanol) are presented in Fig. 2 to understand the influence of environment on the possible causes of crack initiation sites. Fracture surfaces of all the samples were compared with respect to extent of environmental damage during fatigue loading. Fatigue properties of DLC coated and uncoated specimens were shown in Table 1. Uncoated samples in methanol revealed severe damage and pit like formation near the crack initiation site in contrast to coated samples in air as shown in Figs. 2a & b. In addition, numerous secondary cracks were seen along the outer periphery of tested samples. Multiple crack initiation sites resulting in multiple parallel crack fronts are evident in uncoated fatigue tested sample in methanol environment (Fig. 2a). In a similar context, coated samples tested in methanol environment (Fig. 2c) also shown deep pits at the crack initiation site in comparison with uncoated samples tested in methanol environment. However, the fracture surface is smooth and the extent of damage is less severe than uncoated samples.

Adherence of the coating with respect to surface finishing of the substrate, prior to coating was studied for fatigue tested samples. It is clear from Fig. 3 that 1200 grit polished and fatigue tested in air, showed extensive coating delamination and cracks near the crack initiation site. In contrast, very minimal damage is seen in the case of diamond polished samples, even near the crack initiation site which corroborates better adherence of the coating. Thus, better adherence and coating integrity of diamond polished samples resulted in better fatigue performance than 1200 grit polished samples as shown in the Table 1. Possible influence of a high compressive residual stresses on improved fatigue behavior in coated samples tested in air cannot be ruled out.
Summary

Diamond-like coating of 2.5 micron thick was X-ray amorphous in nature as evidenced through grazing incidence X-ray diffraction studies. Residual compressive stress of -1.29 ± 0.1 GPa was induced in the material due to coating process. Severe environmental damage and pit formation at the crack initiation site was observed in uncoated fatigue tested samples in methanol environment. Diamond polished samples prior to DLC coating showed better coating adherence and fatigue performance in air than 1200 grit polished samples.

References