Effect of Interlayer Thickness on Fatigue Behavior in A5052 Aluminum Alloy with Diamond-Like Carbon/Anodic-Oxide Hybrid Coating

Yoshihiko Uematsu1,*, Toshifumi Kakiuchi1, Megumi Adachi2 and Takeshi Shinohara2

1Department of Mechanical Engineering, Gifu University, Gifu 501-1193, Japan
2TOCALO Co., Ltd., Akashi 674-0093, Japan

Anodic oxide layer was formed on A5052 aluminum alloy, and subsequently diamond-like carbon (DLC) was deposited to fabricate DLC/anodic-oxide hybrid coating. Plane bending fatigue tests have been performed using the hybrid-coated specimens with different thicknesses of anodic-oxide interlayer. The interlayer thicknesses were 10 and 50 µm, where the thickness of DLC film was fixed as 3 µm. The specimens without coating, with DLC single layer and with anodic-oxide single layer were also used for comparison. The fatigue strengths of the specimens with anodic-oxide single layer were lower than those of the substrate without coating because the corner edge cracking or pin-hole defects could be fatigue crack initiation sites. However, when DLC was deposited on the anodic-oxide layer, fatigue strengths were improved. The hybrid-coated specimens with the interlayer thickness of 10 µm had higher fatigue limit than the substrate and specimens with the interlayer thickness of 50 µm. Thin DLC layer with the thickness of 3 µm could have suppressed the cracking in interlayer, and led to the improvement of fatigue strengths than the substrate. When the interlayer became relatively thicker compared with the DLC film, such as 50 µm, DLC film could not suppress the cracking from the corner edge defects, resulting in the lower fatigue strengths in spite of the presence of hybrid coating. [doi:10.2320/matertrans.M2015302]

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1. Introduction

Aluminum (Al) alloys are attractive as light weight structural materials, and thus the improvement of their fatigue strengths is important in the engineering point of view. It is well known that hard coatings on the material surface could improve fatigue strengths, and DLC film is one of the candidates of hard coating improving fatigue strengths in light weight alloys.1–3) However, the large gap of elastic modulus between hard DLC film and soft substrate leads to the easy delamination of DLC film when the coated material is subjected to relatively high contact pressure.3) The authors had developed DLC/WC-12Co hybrid coating on Al4,5) or magnesium (Mg)6) substrate, in which WC-12Co was thermally sprayed as an interlayer and subsequently DLC was deposited on it. It had been reported that the resistance against delamination under the contact pressure and fatigue strengths were improved by DLC/WC-12Co hybrid coatings. Thus some combinations of hybrid coatings had been investigated. For example, G. Bolelli et al. fabricated DLC/WC-Co hybrid coating on steel and Al plates, and revealed the enhanced wear resistances.7,8) T. Morita et al. had developed DLC/plasma carburizing9) or DLC/plasma nitriding10) hybrid coatings on stainless steel. It had been proved that the friction-wear characteristics and fatigue strengths were improved by those hybrid coatings. The combination of plasma nitriding and fine-particle bombarding was also tried on Ti-6Al-4V11,12) by T. Morita et al. and on AISI 1045 steel13) by S. Kikuchi et al., where fatigue strengths were enhanced. As mentioned above, the authors had succeeded the enhancement of fatigue strengths in Al and Mg alloys by DLC/thermally-sprayed WC-12Co hybrid coatings. However, the shortcoming of DLC/WC-12Co hybrid coating is high cost of thermal spraying process used for the fabrication of interlayer. In this research, therefore, anodic oxidation was adopted for the fabrication of interlayer, and anodic-oxide interlayer was covered by DLC film. By using conventional anodic oxidation, the fabrication cost of hybrid coating could be largely reduced. To use DLC/anodic-oxide interlayer hybrid coating for the mechanical component, understanding of the effect of hybrid coating on fatigue behavior is important. In the present study, plane bending fatigue tests had been conducted using Al alloy having DLC/anodic-oxide hybrid coatings with different interlayer thickness, and the effect of interlayer thickness on fatigue behavior was investigated.

2. Experimental Procedure

2.1 Material and specimen

The material for the substrate is A5052-O aluminum alloy. The chemical composition is as follows; Mg: 2.57, Fe: 0.26, Cr: 0.18, Si: 0.08, Zn: 0.01, Mn: 0.01, Cu: 0.01, Al: bal. (mass%). The mechanical properties of the material are listed in Table 1. The configuration of plane bending fatigue specimen is shown in Fig. 1. The specimen has shallow notch with the stress concentration factor, Kt, of 1.02. Before the coating processes of DLC deposition or anodizing, the specimen surface was mechanically polished using progressively finer grades of emery paper and finally buff-finished.

2.2 Coatings

The substrate material was anodized by conventional anodizing treatment, namely sulfuric acid anodizing. By

Table 1 Mechanical properties of the material (A5052-O).

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength</td>
<td>221</td>
</tr>
<tr>
<td>0.2% proof</td>
<td>104</td>
</tr>
<tr>
<td>Elastic Modulus</td>
<td>63</td>
</tr>
<tr>
<td>Elongation δ (%)</td>
<td>26</td>
</tr>
<tr>
<td>Vickers Hardness</td>
<td>59</td>
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</tbody>
</table>

*Corresponding author, E-mail: yuematsu@gieu-u.ac.jp
changing the treatment time, the thicknesses of anodic oxidation layer were set to be about 10 or 50 µm. Sealing treatment of anodic-oxide coating was not performed. Subsequently, DLC was deposited on the anodic oxidized interlayer by Plasma-Based Ion Implantation and Deposition (PBIID) techniques. The coating equipment has the cubic chamber which is one meter on a side. Fatigue specimens were degreased by ultrasonic cleaning in acetone prior to DLC deposition. Specimens were hung in the chamber, and DLC covered specimens along with their shape. The processing temperature was 80°C where the film growth rate was about 1 µm per one hour. The thickness of DLC coating was 3 µm. The samples with anodic-oxide or DLC single layer was prepared for comparison. Table 2 summarizes the specimens prepared. For example, the specimen with 50 µm anodic-oxide interlayer and 3 µm DLC coating is denoted as “D3A50” as shown in Table 2.

2.3 Testing procedures

Plane-bending fatigue tests had been conducted using Schenk-type fatigue testing machine made by TOKYO KOKI Co. Ltd. Test frequency, \( f \), was 25 Hz and stress ratio, \( R \), was \(-1\), namely fully reversed bending. The strain gage was mounted on the back surface of specimen shown in Fig. 1, and the stress during fatigue test was monitored directly because Schenk-type fatigue testing machine is bending moment control.

3. Results

3.1 Coating appearance

The surface appearances of coatings in D3, A10, A50, D3A10 and D3A50 specimens are revealed in Figs. 2(a)–2(e), respectively. The surface of DLC single layer (Fig. 2(a)) is quite smooth, while those of anodic-oxide single layers were rough. It should be noted that some pin-holes are clearly recognized in A50 specimen (Fig. 2(c)). The surfaces of hybrid coatings were smoother than those of anodic-oxide single layers, but the roughness of the anodic-oxide interlayer could be still recognized on the surface.

Figure 3 shows the cross section of anodic-oxide single layer with the thickness of 50 µm (A50 specimen). In the magnified view of Fig. 3(b), some pin-hole defects with the size of a few µm are recognized as typically shown by the arrows in the figure. The cross section of hybrid coating (D3A50) is shown in Fig. 4, where DLC film was deposited on the anodic-oxide interlayer, indicating that the hybrid-coating was successfully fabricated.

As shown in Fig. 1, the cross section of the specimen is rectangular having corner edges. Figures 5(a)–5(d) reveal the cross sections of the coatings near the corner edges of the specimens A10, A50, D3A10 and D3A50, respectively. The cracking of anodic-oxide layers was recognized near the edges of all specimens. It should be noted that the cracking is wider in the thicker anodic-oxide layers (Fig. 5(b) and 5(d)). The specimen surface morphologies near the corner edges of the specimens A10 and A50 are indicated in Fig. 6. As shown by the arrow in the macroscopic appearance of A50
(Fig. 6(b)), the cracking exits along the corner edge of the gauge section. The magnified views reveal the crack opening is about 1 µm for A10 (Fig. 6(a)), and 20 µm for A50 (Fig. 6(c)). It is considered that the cracking was formed during anodizing process due to the different thermal expansion coefficients between substrate and anodic-oxide layer. Figure 7 indicates specimen surface morphologies near the corner edges in D3, D3A10 and D3A50 specimens. The edge cracking was not found in DLC single layer (Fig. 7(a) and (b)), while that was recognized in D3A50 (Fig. 7(e) and (f)). It should be noted that such corner edge cracking was found on the cross section of D3A10 as shown in Fig. 5(c), while not on the surface of corner edge (Fig. 7(c) and (d)). Furthermore, the width of cracking is narrower in D3A50 (Fig. 7(f)) than in A50 (Fig. 6(c)). Consequently, it could be concluded that DLC filled the cracking of anodic-oxide interlayer during deposition process, and when the thickness of interlayer was 10 µm, the cracking was fully filled by DLC (Fig. 7(c) and (d)). But when the interlayer becomes thicker, the cracking was not fully filled by DLC, and cracking remained on the specimen edge as shown in Fig. 7(e) and (f).

### 3.2 Fatigue strengths

Figure 8 is the S-N diagram of the materials. Fatigue limit was defined as the run-out stress amplitude at $10^7$ numbers of cycles. Compared with the fatigue strength of substrate material, DLC single layer increased fatigue strength similar to the previous reports by the authors. However, anodic-oxide single layers decreased fatigue limits irrelevant to the thickness, indicating detrimental effect of anodic-oxide coating on fatigue properties. In the finite life region, fatigue lives were shorter in A50 than in A10. The fatigue limit of the specimen with hybrid coating, D3A50, increased compared...
with that of A50 without DLC coating. It indicates that hybrid structure is effective for improving fatigue properties. However, the fatigue limit of D3A50 is still comparable to that of the substrate, and the fatigue lives are shorter in the finite life region (low cycle region). The fatigue limit of D3A10 was the highest among all specimens, while the fatigue lives in the finite life region is shorter than DLC single layered specimen. Consequently, it could be concluded that the fatigue strengths of the specimens with hybrid coatings are strongly affected by the thickness of the interlayer.

3.3 Fatigue crack initiation behavior

Figure 9 is the example of crack initiation sites in the substrate material, revealing typical fatigue crack initiation mechanism in aluminum alloys due to the cyclic slip deformation. Crack initiation sites of DLC single layered specimen, D3, is shown in Fig. 10. As macroscopically shown in Fig. 10(a), fatigue crack initiated on the smooth specimen surface.

To investigate crack initiation behavior in detail, fatigue tests was terminated when the number of cycles reached \(0.5 - 0.9N_f\) where \(N_f\) is number of cycles to failure obtained from the basic S-N diagram (Fig. 8). Fatigue-damaged specimen surface was investigated in detail to find fatigue crack, and subsequently the specimen was statically cut to observe fracture surface. Figure 11 reveals fatigue cracks observed on the specimen surface of A10 specimen and their corresponding fracture surfaces \((N = 0.5N_f)\). It should be noted that two fatigue cracks initiated on the smooth specimen surfaces. On the other hand, fatigue crack initiated at the corner edge in A50 specimen as revealed in Fig. 12 \((N = 0.9N_f)\). The crack was observed on the specimen surface (Fig. 12(a)) and the crack initiation site was identified at the corner edge as shown by macroscopic (Fig. 12(c)) and magnified (Fig. 12(d)) views of the crack initiation site. Figure 12(b) indicates the appearance of crack observed at the corner edge. Based on these investigations, it was concluded that crack initiation at the corner edge was dominant in A50 (Fig. 12), while crack predominantly initiated on the smooth specimen surface in A10 (Fig. 11), revealing the dependence of crack initiation mechanism on the thickness of anodic-oxide layer.

The surface and corner edge views of D3A50 specimen are shown in Figs. 13(a) and 13(b), respectively \((N = 0.9N_f)\). As revealed by the fatigue fracture surface (macroscopic (Fig. 13(c)) and magnified (Fig. 13(d)) views), fatigue crack initiated at the corner edge. Similar to A50 specimens,
crack initiation at the corner edge was dominant in D3A50. In the D3A10 specimens, crack observation procedure on the specimen surface was failed. Thus, the fatigue fracture surfaces at high ($\sigma_a = 152$ MPa) and low ($\sigma_a = 143$ MPa) stress amplitudes are shown in Figs. 14(a) and 14(b), respectively. At high stress amplitude, cracks initiated at the locations shown by the arrows in Fig. 14(a), and crack initiation at the corner edge was recognized similar to D3A50. But at lower stress amplitude, two fatigue cracks initiated on the smooth surface (Fig. 14(b)), indicating the dependence of crack initiation behavior on the stress level and the thickness of interlayer.

4. Discussion

4.1 Specimen with DLC or anodic-oxide single layer (A10 and A50)

As shown in Fig. 8, fatigue strength of substrate was enhanced by DLC single layer. This is because cyclic slip deformation was restricted by hard DLC layer on the specimen surface and crack initiation was retarded as described in the previous researches. However, hard anodic-oxide layers could not improve fatigue strengths of substrate. As shown in the fatigue fracture surfaces (Figs. 11 and 12), crack initiation locations are identified at the corner edge (Fig. 12) or on smooth specimen surface (Fig. 11). Figure 15 shows the S-N diagram similar to Fig. 8, but when crack initiated at the corner edge, the symbol was marked by asterisks "*". It is clear from the figure that crack initiated at the corner edge in A50 and on the smooth surface in A10. The corner edge cracking (Fig. 5) is parallel to the loading.
The detrimental effect of anod-coating in A10 specimens resulted in the lower fatigue strengths. In the specimens A10, the crack opening was relatively small, and crack initiation from pin-hole defects in the substrate. It could be concluded that the crack initiation from corner edge was recognized on the fracture surface, the lower fatigue strengths than the substrate. However, anodic-oxide single layer had small pin-hole defects in itself as shown in Fig. 3. Consequently, pin-hole defects in anodic-oxide layer rested in the cracking of the coating in the early stage of fatigue life and lower fatigue strengths than the substrate. It could be concluded that the crack initiation from corner edge cracking in A50 and that from pin-hole defects in coating in A10 specimens resulted in the lower fatigue strength than the substrate. The detrimental effect of anodizing on fatigue properties was also reported by E. Cirik et al. and A. Inoue et al., where the lower fatigue strengths of anodized specimens were attributed to the defects in the coatings.

4.2 Specimen with DLC/anodic-oxide hybrid coating (D3A10 and D3A50)

As revealed in Fig. 15, when the crack initiation at the corner edge was recognized on the fracture surface, the results were identified by adding asterisk marks. It should be noted that fatigue cracks initiated at the corner edge in D3A50 at all stress amplitudes. It is considered that the additive DLC film on anodic-oxide layer could retard crack initiation resulting in the higher fatigue strengths in D3A50 than in A50. However, the detrimental effect of the edge cracking as a crack starter could not be fully eliminated by DLC film over anodic-oxide layer, and thus fatigue limit was still comparable to the substrate. At higher load levels, the earlier crack initiation at the corner edge resulted in the shorter fatigue lives than the substrate.

In D3A10 specimen, however, the dependence of fatigue crack initiation on stress levels could be recognized, where crack initiation at the corner edge was dominant at higher load levels, while on the smooth specimen surface at lower load levels. It should be noted that when crack initiated on the specimen surface, fatigue limit was higher and fatigue lives were longer than the substrate. In A10 specimens without DLC over-layer, lower fatigue strengths than the substrate was attributed to the pin-hole defects in anodic-oxide layer. Thus it could be concluded that DLC film could suppress the cracking in the anodic-oxide layer, and resulted in the highest fatigue strengths in the high-cycle (long life) region. However, at higher load levels than 145 MPa, the stress concentration at the corner edge crack was not negligible, resulting in the lower fatigue strengths than D3 specimen with DLC single layer.

It could be concluded that the corner edge cracking could be introduced when the aluminum component has edges, which has detrimental effect on fatigue strength. But when the thickness of anodic-oxide layer was 10 µm, DLC over layer with the thickness of 3 µm could suppress both crack initiation from corner edge and cracking of anodic-oxide layer, and resulted in the higher fatigue strength than the substrate. It should be noted DLC/anodic-oxide hybrid coating with optimal interlayer thickness could further improve fatigue limit than DLC single layer.

5. Conclusions

DLC/anodic-oxide hybrid coating was fabricated on A5052 alloy with changing the thickness of anodic-oxide interlayer. Plane bending fatigue tests had been conducted to investigate the effect of interlayer thickness on the fatigue behavior. Based on the experimental results, the following conclusions can be made:

(1) DLC single layer could improve fatigue strength of the substrate. However, anodic-oxide single layer had detrimental effects on fatigue properties irrelevant to the thickness, due to the fatigue crack initiation from corner edge crack or pin-hole defects in the layer.

(2) DLC film over anodic-oxide layer could suppress the cracking of anodic-oxide interlayer. The specimens with hybrid coating exhibited higher fatigue strengths than those with anodic-oxide single layer.

(3) When the anodic-oxide layer was thick (50 µm), DLC film could not suppress the cracking from the corner edge. But when the thickness of interlayer was 10 µm, DLC film could suppress the cracking of interlayer, resulting in the higher fatigue strengths than the substrate and specimen with DLC single layer.

(4) DLC/anodic-oxide hybrid coating was effective for improving fatigue strengths and fatigue limit of the substrate, when the interlayer thickness was thin and the applied load level was low. However, if the applied loads were increased, fatigue lives could be shorter than the substrate due to the stress concentration at the corner edge cracking.

REFERENCES

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