Fatigue behavior of A5052 aluminum alloy with diamond-like carbon/electroless nickel plating hybrid coating

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Abstract
Electroless nickel plating was formed on A5052 aluminum alloy, and subsequently diamond-like carbon (DLC) was deposited on nickel plating to fabricate DLC/nickel plating hybrid coating. Cantilever rotating bending fatigue tests were conducted using the specimens with and without coatings. The fatigue strengths of the specimens with DLC or nickel plating single layers were higher than those of the substrate without coating. However, the specimens with nickel plating had large scatter in the fatigue strengths due to some cracking in the coatings. When DLC was deposited on the nickel-plating layer, the fatigue strengths were further improved compared with the specimens with DLC or nickel plating single layers. The observation of fatigue fracture surfaces revealed that fatigue crack initiated at the substrate just beneath the coating in the specimens with DLC or nickel plating single layers. However, subsurface fatigue crack initiation occurred in high cycle fatigue regime of the specimen with hybrid coating. It indicated that the improvement of fatigue strengths could be attributed to the suppression of fatigue crack initiation by coatings, where hybrid coating was more efficient than DLC or nickel plating single layers.

Keywords: Fatigue, Diamond-like carbon, Electroless nickel plating, Hybrid coating, Subsurface crack initiation

1. Introduction

Aluminum (Al) alloys are generally used for load-bearing mechanical components because of their lighter weight and higher specific strengths than conventional steels. Therefore, increasing fatigue strengths of Al alloys is an important issue from the viewpoint of structural reliability and life-time cost saving. It is known that surface treatment is one of the effective methods to improve fatigue strengths of structural alloys. For example, mechanical peening processes such as shot peening (Masaki et al., 2003), laser peening (Masaki et al., 2007) and cavitation peening (Takahashi et al., 2018) could increase fatigue strengths of Al alloys, by increasing surface hardness, giving compressive residual stress and etc. However, such mechanical peening process deteriorate surface accuracy due to the severe plastic deformation induced by shot materials. It is considered that the application of hard coating on the substrate could also improve fatigue strengths without plastic deformation on the specimen surface. For example, anodizing is a popular way to give hard coating on Al substrate. However, anodic-oxide layer on Al substrate sometimes has detrimental effect on the fatigue performances because stress concentration occurs at some defects in the coatings (Shiozawa et al., 2000, Inoue et al., 2012). Recently, diamond-like carbon (DLC) attracts attention as hard coating on Al alloys, because defects in DLC coating are much less than those in anodic-oxide layer. The authors have reported that DLC coating on A5053 Al alloy could successfully improve fatigue strengths of substrate (Uematsu et al., 2015). However, it is well known that the elastic modulus of Al alloys is much lower than that of DLC. Therefore, delamination could easily occur between DLC coating and Al substrate under contact force or steep stress gradient at sharp notches, because DLC coating with higher elastic modulus cannot follow the large deformation of the substrate with lower elastic modulus. The hybrid coating system is one of the candidates to enhance the performance of DLC coating on soft substrate, in which the layer having intermediate properties...
is inserted between DLC coating and soft substrate. The authors had reported about the fabrication of hybrid coatings on Al (Kakiuchi et al., 2013) or magnesium (Mg) (Okada et al., 2010) substrates, in which WC-12Co was thermally sprayed as an interlayer prior to DLC deposition. The hybrid coatings could further improve fatigue strengths of substrates than DLC single coating, and increase the resistance against delamination under the contact pressure. G. Bolelli et al. (2011) had also fabricated DLC/WC-Co hybrid coating on steel and Al plates, and revealed the enhanced wear resistances. However, thermal spraying followed by DLC deposition highly increases the processing cost mainly due the thermal spraying process, because the spraying needs complicated facilities and the material cost of spraying powder is high. In the previous study, therefore, anodization was employed as the interlayer of DLC/anodic-oxide hybrid coating (Uematsu et al., 2015), because anodization process was much cheaper than thermal spraying. However, anodizing layer included porosities in itself, and the cracking of coating occurred at the corner edge of the specimens. Subsequently, simple electroless nickel plating is used as the interlayer in the present study to form DLC/nickel plating hybrid coating, because nickel plating is much cheaper than spraying and the defects in the coating might be less than anodizing.

In this research, rotating bending fatigue tests were performed using Al alloy, A5052 with DLC/nickel plating hybrid coating to investigate the effect of hybrid coating on the fatigue performances under stress gradient. The test results were compared with those of the DLC or nickel-plating single-layered specimens and the substrate to discuss the different effect among three coatings.

2. Experimental procedure

2.1 Specimens

The substrate material is A5052 BD-H34 Al alloy, which was extruded followed by stabilization at 150 °C (423 K). The chemical composition is as follows; Mg: 2.5, Fe: 0.14, Cr: 0.24, Si: 0.06, Zn: 0.01, Mn: 0.02, Cu: 0.01, Al: bal. (wt%). The mechanical properties of the substrate material are summarized in Table 1. Figure 1 show the fatigue specimen configuration with the minimum diameter of 6 mm. Before the coating processes of DLC deposition or anodizing, the specimen surface was mechanically polished using #2000 grade emery paper and finally buff-finished to obtain mirror surface.

The electroless nickel plating (Ni plating) was formed on the substrate material with the thickness of approximately 20 μm. In the previous cases of DLC/WC-Co (Kakiuchi et al., 2013, Okada et al., 2010) or DLC/anodic-oxide (Uematsu et al., 2015) hybrid coatings, the thickness of the interlayers varied in the range of 10 ~ 80 μm. Thus the thickness of the plating in the present study was set to be 20 μm for comparison. Subsequently, DLC was deposited on the Ni plating by a Plasma-Based Ion Implantation and Deposition (PBIID) technique. The processing temperature was 150 °C (423 K) and the thickness of DLC coating was 3 μm. It should be noted that the processing temperature is the same with that of the stabilization, indicating that the temperature rise during DLC deposition has small effect on the mechanical properties.

| Tensile strength $\sigma_0$ (MPa) | 0.2% proof $\sigma_{0.2}$ (MPa) | Elastic Modulus $E$ (GPa) | Elongation $\delta$ (%) | Vickers Hardness ($HV$) |
|----------------------------------|---------------------------------|---------------------------|------------------------|------------------------|
| 265                              | 257                             | 71                        | 13                     | 83                     |

Fig. 1 Rotating bending fatigue specimen configuration.
of the substrate material. Hereafter, the specimens with DLC/nickel plating hybrid coating would be denoted as “D3N20” specimens in the figures. The single-layered specimens having Ni plating with the thickness of 20 μm or DLC coating with the thickness 3 μm were also fabricated as reference materials, which would be denoted as “Ni20” and “D3” specimens, respectively. Consequently, four kinds of specimens, namely D3Ni20, D3, Ni20 and substrate were used for the fatigue tests. The macroscopic appearances of four kinds of specimens are shown in Fig. 2.

2.2 Testing procedures

Cantilever rotating bending fatigue tests were conducted using the samples shown in Fig. 2. The test frequency, \( f \), was 19 Hz and stress ratio, \( R \), was -1. The mirror-finished specimens were used as the substrate material, and the other coated materials were used under as-coated condition. The detail of coating morphology and fatigue fracture surfaces were observed by a scanning electron microscope (SEM).

3. Results and discussion

3.1 Coating appearances

The surface appearances of D3, Ni20 and D3Ni20 specimens are revealed in Figs. 3~5, respectively. The DLC single-layered specimen (Fig. 3) has smooth surface, while some pin-hole defects are found as shown in the magnified view of Fig. 3(b). It has been reported that corrosion fatigue strengths largely decreased due to the penetration of corrosive environment through this kind of pin-hole defects of DLC film (Uematsu et al, 2010). Figure 4 shows the surface morphology of Ni-plating single-layered specimen, which macroscopically has smooth surface (Fig. 4(a)). However, micro cleavages are found in the microscopic observation (Fig. 4(b)). Figure 4(c) is the tilted SEM view of the specimen surface. It reveals that the specimen surface is covered by small spheroidal projections. The surfaces of the hybrid coating are shown in Fig. 5. The micro-cleavages in Ni-plating single-layered specimen (Fig. 4(b)) are not found in the hybrid coating.

![Fig. 2 Macroscopic appearances of specimens: (a) Substrate, (b) D3, (c) Ni20, (d) D3Ni20.](image)

![Fig. 3 Surface morphology of DLC single-layered specimen (D3): (a) Macroscopic view, (b) Magnified view. Small defect is seen in the DLC coating.](image)
Fig. 4 Surface morphology of Ni-plating single-layered specimen (Ni20): (a) Macroscopic view, (b) Magnified view, (c) Tilted view. Micro-cleavages are seen in the Ni plating.

Fig. 5 Surface morphology of DLC/Ni-plating double-layered specimen (D3Ni20): (a) Macroscopic view, (b) Magnified view, (c) Tilted view, (d) Cross section. DLC is deposited on Ni plating interlayer. Micro-cleavages are not seen in the magnified views of the surface and cross section.
coating as shown in Fig. 5(b). Figure 5(c) is a tilted view showing small spheroidal projections induced by DLC deposition. The cross section of the hybrid coating is revealed in Fig. 5(d). DLC layer is successfully deposited on the Ni plating. It should be noted that the Ni-plating interlayer is dense without cracking, revealing that the cleavages seen in Fig. 4(b) did not penetrate into the thickness direction of Ni plating.

3.2 Fatigue test results

The S-N curves of the substrate and coated specimens are shown in Fig. 6. Fatigue limit is defined as the run-out stress amplitude at $10^7$ cycles. The substrate material has the lowest fatigue strengths, in which the fatigue limit is within 145-150 MPa. Compared with the substrate, DLC single layer increased fatigue strengths similar to the previous report by the authors (Uematsu et al., 2015). The fatigue limit is within 160-180 MPa for the DLC single-layered specimens. Ni plating single layer could also increase fatigue strengths, where the fatigue limit is within 165-170 MPa. However, it should be noted that large scatter is seen in the fatigue strengths of the Ni-plating single-layered specimens. One sample ran out at $10^7$ cycles at the stress amplitude of 190 MPa, while another sample failed at $2.7 \times 10^4$ cycles at the stress amplitude of 170 MPa. It is assumed that some cleavages seen in the Ni-plating single-layered specimen (Fig. 4(b)) are large enough to affect the fatigue strengths, resulting in the large scatter. However, such large scatter in the fatigue strengths is not seen in the DLC single-layered specimens. It indicates that the pin-hole defects seen in DLC film (Fig. 3(b)) have little effect on the scatter of fatigue strengths, while smaller pin-hole defects would result in the higher fatigue performances (Uematsu et al., 2011). The DLC/Ni plating hybrid-coated specimens have the highest fatigue strengths. The fatigue limit is within 200-210 MPa, indicating that the fatigue limit was improved about 25~38% compared with the substrate. However, one sample failed at $4.8 \times 10^4$ cycles at the stress amplitude of 210 MPa. It implies that some large cracking in Ni-plating could have effect on the fatigue strengths even in the hybrid-coated samples.

In Ni-plating single-layered specimens, hydrogen diffusion into the substrate could occur during the plating process. And the temperature rise during DLC deposition (150 °C) is similar to the baking procedure removing hydrogen in the substrate. Therefore, hydrogen embrittlement (Ohnishi, 1989) could be one of the reasons of the different fatigue strengths between hybrid-coated and Ni-plating single-layered specimens, because hydrogen content in hybrid-coated specimen might be lower than that in Ni-plating single-layered specimen. However, hybrid-coated specimens have higher fatigue strengths than DLC single-layered specimens, indicating that the hybrid coating is effective from the viewpoint of the suppression of cyclic slip deformation in the substrate.

![Fig. 6 S-N diagram. Arrows indicate run-out samples. DLC/Ni plating hybrid coated specimen has the highest fatigue strengths.](image)

3.3 Fractographic analyses

Figure 7 shows typical fatigue fracture surface near the crack initiation site of Ni-plating single-layered specimen.
The ratchet marks on the fracture surface seen in Fig. 7(a) reveal multiple fatigue crack initiation. As shown in the arrows in Fig. 7(b), some cracks are recognized in the coating. However, the cracking in the coating could not be directly related to the fatigue crack initiation, because the number of the cracking in the coating was less than the number of multiple crack initiation sites estimated from the ratchet marks. It should be noted that the Ni plating in Fig. 7(c) is dense without micro-cracks, indicating that most of the micro-cleavages shown in Fig. 4(b) did not reach the substrate as described in the section 3.1.

Figure 8 indicates the fatigue fracture surfaces of the hybrid-coated specimen, which had the shortest fatigue life of $4.8 \times 10^4$ cycles. One of the crack initiation sites is shown in Fig. 8(b). The cracking of interlayer is recognized similar to Ni-plating single-layered specimen (Figs. 7(a) and (b)). However, the cracking could not be simply correlated with the shortest fatigue life because similar cracking of the interlayer was also recognized in the hybrid-coated specimen failed in the high cycle fatigue regime as will be mentioned below. Figure 9 is the crack initiation site of the specimen with hybrid coating, which failed in the high cycle fatigue regime at $3.6 \times 10^6$ cycles. Similar to Ni-plating single-layered specimen (Fig. 7(a)), many ratchet marks are seen, indicating multiple crack initiation. Cracking of the coating is also recognized. Figure 9(b) is the magnified view of one of the crack initiation sites indicated by the arrow in Fig. 9(a). As shown in Fig. 9(b), subsurface crack initiation had occurred, where crack initiation site located at about 100 μm beneath the specimen surface. Figure 9(c) is the tilted view of the subsurface crack initiation site, in which the arrow indicates the crack origin. This subsurface crack initiation represents the characteristic feature of the high cycle fatigue fracture of the hybrid-coated specimen. It implies that the hybrid coating could suppress fatigue crack initiation at the substrate surface enough to induce subsurface crack initiation, while DLC and Ni-plating single layers could not induce subsurface crack initiation. It could be concluded that the high fatigue strengths in the hybrid-coated specimens were attributed to the suppression of cyclic slip deformation in the substrate, namely the suppression of fatigue crack initiation by the hybrid coating, which had better effect on fatigue strengths than DLC single coating. However, it should be noted that hybrid-coating is not effective if the Ni-plating layer has large cracking in itself. The fractographic analyses of DLC single-layered sample could be found elsewhere (Uematsu et al., 2010, Uematsu et al., 2015).

![Fig. 7 SEM micrographs showing fatigue fracture surfaces near crack initiation site in Ni20 specimen ($\sigma_a = 230$ MPa, $N_f = 7.8 \times 10^3$): (a) General view, (b) Low magnification, (c) High magnification. Arrows indicate the cracking of coating. Many ratchet marks represent multiple fatigue crack initiation.](image)
4. Conclusions

DLC/electroless nickel plating hybrid coating was fabricated on A5052 alloy substrate, in which DLC was deposited on the Ni-plating interlayer. Rotating bending fatigue tests had been conducted to investigate the effect of hybrid coating on the fatigue behavior. Based on the experimental results, the following conclusions can be made:

Fig. 8. SEM micrographs showing fatigue fracture surfaces near crack initiation site in D3Ni20 specimen ($\sigma_a = 210$ MPa, $N_f = 4.8 \times 10^4$): (a) General view, (b) Magnified view at crack initiation site shown by the black arrow in Fig. (a).

Fig. 9. SEM micrographs showing fatigue fracture surfaces near crack initiation site in D3Ni20 specimen ($\sigma_a = 220$ MPa, $N_f = 3.8 \times 10^4$): (a) General view, (b) Magnified view at crack initiation site shown by the black arrow in Fig. (a), (c) Tilted view of Fig. (b). White arrows indicate subsurface crack initiation sites.
(1) Both DLC and Ni-plating single layers could improve fatigue strengths of the substrate. However, Ni-plating single-layered specimens had large scatter in the fatigue strengths due to some cracking in Ni-plating.

(2) DLC/Ni-plating hybrid coating could further improve fatigue strengths compared with DLC or Ni-plating single layers. However, one hybrid-coated sample failed at short fatigue life, indicating that the scatter in fatigue life still existed.

(3) Subsurface crack initiation occurred in DLC/Ni-plating hybrid-coated specimen in high cycle fatigue regime. Hybrid coating could suppress fatigue crack initiation in the substrate, resulting in the high fatigue strengths of the hybrid-coated specimens.

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