Effect of Hybrid Surface Treatments on Fretting Fatigue Strength of Stainless Steel

Toshihiro Omori1,*, Tatsuro Morita2, Kohei Okada2 and Hideaki Maeda1

1Department of Research and Development, Torishima Pump Mfg. Co., Ltd., Takatsuki 569-8660, Japan
2Department of Mechanical and System Engineering, Graduate School of Science and Technology, Kyoto Institute of Technology, Kyoto 606-8585, Japan

This study was conducted to investigate the effect of hybrid surface treatments on the fretting fatigue strength of austenitic stainless steel JIS SUS316. The hybrid surface treatments were composed of plasma nitriding (hereafter, PN) and fine particle bombarding (FPB) or PN and diamond-like carbon (DLC) coating. Although the combination of PN and FPB slightly increased the friction coefficient, it improved wear resistance through the formation of a hardened layer. The combination of PN and DLC coating markedly improved the friction-wear properties because the thin DLC layer was supported by the hardened layer formed below it. Neither treatment had any influence on mechanical properties. The above hybrid surface treatments greatly improved the fretting fatigue strength. The improvement percentage was 44% in the case of the combination of PN and FPB, and reached 54% with the combination of PN and DLC coating. To examine corrosion resistance, the hybrid-surface-treated materials were fully immersed into 5% salt water held at 363 K (90 degrees C). The results showed that their corrosion resistance was maintained for 2.42 Ms (28 days) without surface damage. [doi:10.2320/matertrans.M2014358]

(Received October 10, 2014; Accepted December 5, 2014; Published January 23, 2015)

Keywords: fretting fatigue strength, stainless steel, hybrid surface treatment, wear resistance, mechanical properties, corrosion resistance

1. Introduction

Fretting is a special kind of wear induced by repeated relative surface motion. This phenomenon often occurs on the contact surfaces of combined machine parts and causes marked damage. If fretting is induced under applied cyclic stress, it facilitates crack initiation and greatly decreases fatigue strength.1–4) Accordingly, the above phenomenon, called fretting fatigue, has to be adequately prevented to assure the safety of rotating machine products such as turbines and water pumps.

Fretting fatigue is complicated because it is related to many factors such as friction-wear, metal fatigue, complex stress conditions at contact surfaces and their interaction.5–8) However, fretting fatigue should be preventable by simultaneous improvements in the friction-wear properties and the fatigue strength of metals. The above improvements can be achieved by appropriate surface treatments. Moreover, surface treatments offer an engineering advantage in that there is no need to correct the shapes of machine parts.

In recent years, one of the authors has investigated the effects of various surface treatments such as plasma hardening treatments,9,10) fine-particle bombarding (hereafter, FPB),11–13) diamond-like carbon (DLC) coating9,14) and their combinations as hybrid surface treatments.9,10,15,16) The results showed that hybrid surface treatments were more effective for improving the friction-wear properties and the fatigue strength than individual treatments.

From the above background, this study was conducted to investigate the effect of hybrid surface treatments on fretting fatigue strength. The hybrid surface treatments were composed of plasma nitriding (hereafter, PN) and FPB or PN and DLC coating. Since those treatments were intended for use in the machine parts of water-pumps, typical austenitic stainless steel JIS SUS316 was selected as the substrate.

| Table 1 Chemical compositions of stainless steel JIS SUS316 used for: (a) specimens shown in Fig. 1(b) and (c); (b) specimens shown in Fig. 1(a) and (d). |
|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
|                  | C    | Si    | Mn    | P    | S    | Ni    | Cr    | Mo    | Fe    |
| (a) (mass%)      |      |      |      |      |      |      |      |      |      |
|                  | 0.05 | 0.23 | 1.30  | 0.04 | 0.03 | 10.00 | 16.95 | 2.00  | Bal.  |
| (b) (mass%)      |      |      |      |      |      |      |      |      |      |
|                  | 0.05 | 0.21 | 1.34  | 0.03 | 0.02 | 10.02 | 16.88 | 2.00  | Bal.  |

For comparison, we prepared the solution-treated material and materials on which the individual surface treatments were performed, respectively. For all materials, we comprehensively examined the characteristics of the formed layers, friction-wear properties, mechanical properties, fretting fatigue strength and corrosion resistance. The fretting fatigue was evaluated by a testing method newly designed for this study.

2. Materials and Experimental Procedures

2.1 Materials and treatments

Table 1 shows the chemical compositions of the austenitic stainless steel JIS SUS316 used in this study. The material shown in Table 1(a) was round-bars with a diameter of 5 mm, and the material shown in Table 1(b) was round-bars with a diameter of 12 mm. Table 2 shows the treatment conditions. Before machining, the above materials were solution-treated under the conditions shown in Table 2. Hereafter, the solution-treated material is called the ST material.

Figure 1 shows the specimen shapes. The material shown in Table 1(a) was machined to the shapes given in Fig. 1(b) and (c). The fatigue specimens (Fig. 1(b)) were used to...
obtain the fatigue strength of the ST material. The round-bar specimens (Fig. 1(c)) were used for the investigation of microstructures, observation of DLC layers and measurement of hardness distributions as well as for the tensile and fretting fatigue tests. The material shown in Table 2 was machined to make the button specimens (Fig. 1(a)) and the contact pads (Fig. 1(d)). The button specimens were used to investigate the surface features, friction-wear properties and corrosion resistance. The contact pads were used for the fretting fatigue test.

The test sections of all specimens except the contact pads were polished to mirror surfaces with emery papers and alumina powders. Then, PN, FPB and DLC coatings were applied under the conditions shown in Table 2. The hybrid surface treatment was composed of PN and FPB (PN/FPB) or PN and DLC coating (PN/DLC). Hereafter, the abbreviations of the above treatments are also used to refer to the materials. In the PN/DLC material, the specimen surfaces were polished again to mirror surfaces before DLC coating. No surface treatment was performed for the contact pads (surface roughness $R_a = 0.8 \, \mu m$).

### 2.2 Experimental procedures

The surface features were observed by scanning electron microscopy (SEM). The following experiments were conducted on the cross sections polished to mirror surfaces. The microstructures near the surfaces were investigated by electron back-scattered diffraction (EBSD) analysis. The DLC layers, generated on the surfaces of the DLC and PN/DLC materials, were observed by SEM. The hardness distributions from the surfaces to the inside were obtained by the micro-Vickers hardness test. Hardness was measured three times at each depth and the averages were used as the data.

The friction-wear test was performed under the conditions shown in Table 3, using a ball-on-disc testing machine. For the ST, FPB, PN and PN/FPB materials, the tests were conducted until a sliding distance of 100 m. Since the DLC and PN/DLC materials possessed good wear resistance, the tests were performed until a sliding distance of 2200 m. After the tests, the wear tracks were observed by SEM. At the same positions, the distributions of iron, oxygen and carbon were investigated by energy dispersive X-ray spectroscopy (EDS) analysis.

The tensile test was conducted at room temperature in air, based on JIS Z 2241. Strain was measured by strain gauges pasted onto the specimen surfaces. Three specimens were tested for each material and the averages were used as the data. After the tests, the fracture surfaces were observed by SEM.

The fretting fatigue was evaluated by a method newly designed for this study. Figure 2 shows an explanation of this test. The testing method was as follows: A specimen (Fig. 1(c)) was firstly sandwiched by a pair of half-cylindrical contact pads (Fig. 1(d)). They were inserted together into the collet of the rotating-bending fatigue testing machine (cantilever type) shown in Fig. 2. After the side surfaces of the two contact pads were precisely aligned, the collet was tightened under a torque controlled by a torque wrench and contact stress was applied on the specimen surface. Arbitrary bending stress was applied by hanging weights at the opposite side to the clamped side. Finally, the specimen was rotated at room temperature in air at a rotating speed of 50 Hz (stress ratio $R = -1$).
In the above test, a new pair of contact pads was used for every specimen. The contact stress distribution was calculated by three-dimensional finite-element method (Marc produced by MSC Software). The calculation result showed that the contact stress was 87 MPa at the middle point of the contact pads. After the tests, the crack initiation sites were observed by SEM.

The above fatigue testing machine was also used to obtain the fatigue strength of the ST material (Fig. 1(b)). This test was conducted at a rotating speed of 50 Hz (stress ratio \( R = -1 \)).

Corrosion resistance was investigated by fully immersing the button-specimens into 5% salt water held at 363 K (90 degrees C) for 2.42 Ms (28 days). The salt water was replaced after 14 days to avoid degradation. Although “day” is not an SI unit, it was used to ease understanding. The mass of the specimens was measured after 0, 7, 14 and 28 days by a precision electronic scale. At the same time, the surface features were observed by SEM.

3. Experimental Results and Discussion

3.1 Surface properties and hardness distributions

Figure 3 shows the surface features of all materials and their microstructures (IPF maps) investigated near the
surfaces. Figure 4 shows the features of the DLC layers generated on the DLC and PN/DLC materials. Figure 5 shows the hardness distributions measured from the surfaces to the inside.

As shown in Fig. 3(a), the surface of the PN materials was smooth. The surface roughness of the FPB material was relatively large because the collision of fine-particles formed a number of small craters. The DLC material possessed a smooth surface, as did the ST material.

The surface of the PN material was slightly roughened by the treatment. On the surface of the PN/FPB material, a number of craters were observed; however, their depth was relatively shallow because the hardened layer was formed before FPB. In the PN/DLC material, the surface was polished again before DLC coating, so its surface was smooth, as was that of the DLC material.

FPB affected only the region near the surface. Moreover, the PN and DLC coatings were conducted at relatively low temperatures (Table 2). As a result, the microstructures in the substrates of all surface-treated materials were almost the same as those of the ST material (Fig. 3(b)).

As understood from Fig. 4, DLC coating generated surface layers of thickness 1.5 µm on the DLC and PN/DLC materials. Each surface layer was composed of two layers; that is, the outermost layer was a single DLC layer and the second layer was an intermediate layer to improve adhesion by continuous change in composition. Hereafter, the above surface layers are simply called the DLC layer.

In our previous study, DLC coating was performed on stainless steel under the same conditions used in this study. For reference, the characteristics of the DLC layer obtained in the previous study are shown below: thickness 1.4 µm, Young\'s modulus 300 GPa, hardness 66 GPa, adhesion force 45 N. Here, the thickness of the DLC layer was measured by the spherical drilling method (Calotest). The hardness and Young\’s modulus were measured by the nano-indentation test. The adhesion force was obtained by the scratch test.

As shown in Fig. 5, the hardness distribution of the ST material (○) was constant from the surface to the inside. In the FPB material (▲), although the hardened layer of thickness 40 µm was formed by strain hardening, its hardness level was low. The hardness distribution of the DLC material (■) was almost the same as that of the ST material.

In the PN material (◇), a hardened layer 20 µm in thickness was formed. Although the subsequent FPB increased its thickness (PN/FPB material, ◆), the hardness near the surface decreased. DLC coating had no influence, so the hardness distribution of the PN/DLC material (□) was the same as that of the PN material.

### 3.2 Friction-wear properties

Figure 6 shows the relationship between sliding distance and friction coefficients. Figure 7 shows the features of the wear tracks observed after the tests and the results of the EDS analysis.
As shown in Fig. 6(a), the friction coefficients of the PN and PN/FPB materials were slightly higher than those of the ST and FPB materials. In general, the friction coefficient is determined by the product of contact area and shear resistance. The formation of the hardened layer by PN decreased the contact area and increased the shear resistance at the same time; however, it seemed that the decrease of contact area was smaller than the increase of shear resistance because a high test force was used in this study (Table 3). As a result, the friction coefficients of the PN and PN/FPB materials became slightly higher than the levels for the ST and FPB materials.

The width of the wear track of the FPB material was the same as that of the ST material (Fig. 7). This result showed that the hardened layer formed by strain hardening through FPB was easily lost under the high test force and had no improving effect on the wear resistance.

A number of oxide particles were observed on the wear tracks of the PN and PN/FPB materials. These observation results suggested the occurrence of abrasive wear; however, the wear resistance of the PN and PN/FPB materials was greatly improved by the formed hardened layers. If comparing both materials, the wear track of the PN/FPB material was wider than that of the PN material. Although the hardened layer of the PN/FPB material was thicker, the hardness near the surface was lower (Fig. 5). Consequently, the wear resistance of the PN/FPB material was slightly inferior to that of the PN material.

As understood from Fig. 6(b), the friction coefficient of the DLC material was much lower than those of the other materials due to the effect of the DLC layer. The friction coefficient of the PN/DLC material was lower than that of the DLC material although their DLC layers were the same. This very low friction coefficient was thought to be due to the following causes:

The formation of the hardened layer suppressed deformation induced below the DLC layer by the contact. This maintained the flateness of the surface and reduced friction force arising from geometrical change of the contact surface. At the same time, the contact area decreased, so the contact stress increased. This increase of the contact stress facilitated the graphitization of DLC and decreased the friction force. As a result, the PN/DLC materials showed a very low friction coefficient.

The friction coefficient of the DLC material gradually increased from the sliding distance of 1000 m (Fig. 6(b)). As
shown in Fig. 7, the DLC layer was partially lost along the wear track (the black lines in the C distribution) through abrasion and abruption. In such regions, the substrate was exposed (the white lines in the Fe distribution). The above results suggested that the gradual exposure of the substrate caused the increase in the friction coefficient of the DLC material from the sliding distance of 1000 m.

In contrast, the low friction coefficient of the PN/DLC material was maintained until the end of the test, as shown in Fig. 6(b). Although the DLC layer was worn, it uniformly remained with no marked exposure of the substrate (Fig. 7). This uniform wear occurred because the DLC layer was supported by the hardened layer below it. As a result, the PN/DLC material demonstrated excellent friction-wear properties.

### 3.3 Mechanical properties

Table 4 shows the mechanical properties and the grain size of the substrates, fretting fatigue strength and its improvement percentage.

<table>
<thead>
<tr>
<th></th>
<th>Young’s modulus E/GPa</th>
<th>Yield strength σy/MPa</th>
<th>Tensile strength σt/MPa</th>
<th>Elongation (%)</th>
<th>Reduction in area (%)</th>
<th>Grain size d_g/µm</th>
<th>Fretting fatigue strength σwf/MPa</th>
<th>Improvement percentage of σwf (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ST</td>
<td>188</td>
<td>235</td>
<td>591</td>
<td>57</td>
<td>75</td>
<td>15</td>
<td>180</td>
<td>—</td>
</tr>
<tr>
<td>FPB</td>
<td>173</td>
<td>238</td>
<td>593</td>
<td>55</td>
<td>76</td>
<td>15</td>
<td>180</td>
<td>0</td>
</tr>
<tr>
<td>DLC</td>
<td>187</td>
<td>236</td>
<td>591</td>
<td>59</td>
<td>75</td>
<td>16</td>
<td>200</td>
<td>11</td>
</tr>
<tr>
<td>PN</td>
<td>211</td>
<td>236</td>
<td>590</td>
<td>56</td>
<td>75</td>
<td>13</td>
<td>220</td>
<td>22</td>
</tr>
<tr>
<td>PN/FPB</td>
<td>198</td>
<td>241</td>
<td>592</td>
<td>56</td>
<td>75</td>
<td>15</td>
<td>260</td>
<td>44</td>
</tr>
<tr>
<td>PN/DLC</td>
<td>196</td>
<td>235</td>
<td>589</td>
<td>57</td>
<td>75</td>
<td>15</td>
<td>280</td>
<td>56</td>
</tr>
</tbody>
</table>

In contrast, the low friction coefficient of the PN/DLC material was maintained until the end of the test, as shown in Fig. 6(b). Although the DLC layer was worn, it uniformly remained with no marked exposure of the substrate (Fig. 7). This uniform wear occurred because the DLC layer was supported by the hardened layer below it. As a result, the PN/DLC material demonstrated excellent friction-wear properties.

#### 3.4 Fretting fatigue strength

Figure 9 shows the S-N curves obtained by the fretting fatigue test. This figure includes the results of the conventional fatigue test of the ST material. To ease comparison, the fretting fatigue strengths, obtained from Fig. 9, are summarized in Table 4. Figure 10 shows the features of the fretting fatigue fracture surfaces and the side surfaces observed near the crack initiation sites.

As shown in Fig. 9, the fretting fatigue strength of the ST material (180 MPa, ●) was much lower than its conventional fatigue strength (360 MPa, ○). In the ST material, the fretting fatigue crack initiated from the specimen surface at the contact edge (Fig. 10), and fine brown abrasion powders were observed there. The results of the EDS analysis showed these abrasion powders to be ferrous oxides. The above results showed that the occurrence of fretting greatly decreased the fatigue strength of stainless steel.

The fretting fatigue strength of the FPB material (Fig. 9, ▲) was the same as that of the ST material (●). In all surface-treated materials, including the FPB material, the fretting strength were almost the same in all materials. Furthermore, the fracture of the formed surface layers did not propagate to the inside. Consequently, there was no influence on the ductility (elongation and reduction in area) and the tensile fracture surfaces showed ductile features with dimples (Fig. 8).

Table 4  Mechanical properties, grain size of substrates, fretting fatigue strength and its improvement percentage.

![Fig. 8 Features of the tensile fracture surfaces.](image-url)
fatigue cracks initiated from the specimen surfaces at the contact edges. On the contact surface of the FPB material, wear marks were clearly observed near the crack initiation site (Fig. 10). For this material, since the formed hardened layer was easily lost during the test, there was no improvement in the fretting fatigue strength.

The fretting fatigue strength of the DLC material was only slightly increased (Fig. 9, ■), although its friction-wear properties greatly improved (section 3.2). As shown in Fig. 10, the DLC layer was almost lost on the contact surface near the crack initiation site. This result suggested that the durability of the DLC layer was insufficient to increase the fretting fatigue strength under high contact stress.

In the PN material, the formed hardened layer improved the wear resistance, as mentioned in section 3.2. The hardened layer possessed high durability because it was formed by diffusion of nitrogen. Furthermore, PN greatly increased the fatigue strength of stainless steel, as reported in our previous study.10) As a result, the crack initiation was effectively suppressed and the fretting fatigue strength increased (Fig. 9, ○).

It was thought that the above improvement was also related to the introduction of compressive residual stress. However, if nitrogen is diffused at high concentration into austenitic stainless steel, a special phase, called “S phase”, is generated near the surface. This phase possesses the different lattice constants from those of the austenitic phase.17) Although the stress constant of the S phase is needed for

---

**Fig. 9** S-N curves obtained by the fretting fatigue test, including the results of the conventional fatigue test of the ST material.

---

**Fig. 10** Features of the fatigue fracture surfaces and the side surfaces.
X-ray residual stress evaluation, its measurement is difficult. For this reason, no measurement of residual stress could be conducted in this study.

Although the wear resistance of the PN/FPB material was slightly worse than that of the PN material (section 3.2), its fretting fatigue strength was higher (Fig. 9, △). As explained above, we could not measure the residual stress introduced on the PN/FPB material. According to our previous studies, however, FPB after plasma nitriding was effective for increasing the fatigue strength of titanium alloy because FPB introduced high compressive residual stress. Based on the above study, it is reasonable to infer that the marked improvement in the fretting fatigue strength of the PN/FPB material resulted from high compressive residual stress introduced by FPB.

The friction-wear properties of the PN/DLC material were markedly improved because the DLC layer was sufficiently supported by the hardened layer formed by PN (section 3.2). According to our previous study, furthermore, the combination of PN and DLC coating greatly increased the fatigue strength of stainless steel. As a result, the fretting fatigue strength of the PN/DLC material markedly improved (Fig. 9, □) and the improvement percentage reached 56%. In this study, the PN/DLC treatment was the best treatment for increasing the fretting fatigue strength.

3.5 Corrosion resistance

Figure 11 shows the relationship between immersion time and the mass of the specimens. Figure 12 shows the changes in surface features of each material.

As shown in Fig. 11, there was no change in the mass of all materials at 28 days. Moreover, no marked changes were observed on their surfaces by the end of the test. The above results indicate that no marked deterioration of the corrosion resistance was induced in the range of the test period by the surface treatments investigated in this study.

4. Conclusions

(1) The hybrid surface treatment composed of PN and FPB formed a hardened layer with a thickness of 40 µm.

(2) The hybrid surface treatment composed of PN and DLC coatings formed a DLC layer with a thickness of 1.5 µm and a hardened layer with a thickness of 20 µm below it. The friction-wear properties markedly improved because the thin DLC layer was supported by the hardened layer below it.

(3) The above treatments had no influence on the micro-structure of the substrate. Accordingly, there were no changes in the mechanical properties.

(4) The hybrid surface treatments greatly improved the...
fretting fatigue strength. The improvement percentage was 44% in the case of the combination of PN and FPB, and it reached 56% in the combination of PN and DLC coating.

(5) The corrosion resistance of the hybrid-surface-treated materials was maintained in 5% salt water held at 363 K (90 degrees C) for 2.42 Ms (28 days) without marked surface damage.

REFERENCES