Double fine particle peening to create tribological surface
Enriched with carbon-black and diamond

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**Introduction**
Fine particle peening (FPP) is an advanced peening technology where the surfaces are bombarded by finer shot particles than conventional shot peening. FPP has been a practical technology to improve fatigue properties and tribological properties of the mechanical components. In addition to that, FPP has an alternative potential because it can transfer shot particle elements onto the treated surfaces [1]. By enriching surfaces with the shot particle elements, FPP can provide various effects such as enhancing coating adherence [2], improving biocompatibility [3] and so on. Thus, material transfer induced by FPP possibly plays a key role to create modified surfaces.

To enhance benefits from FPP, the authors have been developed special fine particles to be transferred onto the FPP-treated surfaces. Transfer induced by FPP can be accelerated by employing hybridized particles that consist a core particle and overlying material to be transferred [4]. For tribological application, two types of hybridized particles have been proposed: carbon-black/steel (CBS) particle and diamond/steel (DS) one. FPP using the CBS hybridized particle can reduce and stabilize friction coefficient of the treated surface because transferred carbon-black acts as a lubricant [5]. FPP using the DS hybridized particle embeds diamond grains on the treated surface, increasing wear resistance [6]. It is supposed that reduced friction coefficient as well as enhanced wear resistance are simultaneously achieved by enriching the surface with both of carbon-black and diamond.

**Objectives**
In the present study, the authors attempted a double fine particle peening (double-FPP) to create a modified surface enriched with both of carbon-black and diamond. The proposed process consists of two steps: FPP using the CBS particle and that using the DS one. Each step aims to transfer carbon-black and diamond, respectively. Double shot peening has been a common technique in order to improve fatigue properties by optimizing residual stress distribution and surface roughness [7,8]. However, the proposed double-FPP is oriented to modify the surface by transferring particle elements; the goal is to concentrate two different substances in one surface.

The aim of this study is to clarify the effects of the double-FPP on the transfer phenomena as well as tribological characteristics of the treated surfaces. Transfer of carbon-black and diamond during the double-FPP was carefully analysed, and its roles on the friction and wear were discussed. An appropriate sequence of the double-FPP was explored.

**Methodology**
Two types of hybridized particles, the CBS particle and the DS particle, were prepared to employ for the double-FPP. For the CBS particle, commercial carbon-black powder and steel particle of 70\(\mu\)m in diameter (Fig.1 (a)) were mixed by agitation for 60 minutes in mechanical mortar. The mass ratio of the carbon-black and the steel particle was 1:99. After agitation, carbon-black covers on the surface of the steel particle as shown in Fig.1 (b). To create the DS particle, ball-milling was performed by using a planetary ball mill. Diamond grains of 1\(\mu\)m in diameter and the 70\(\mu\)m steel particle were mixed with a mass ratio of 1:99 and then milled in a zirconia vessel for 15 minutes. The rotational speed of vessel was 2000rpm. Fig.1 (c) shows typical feature of the particle after ball-milling. Diamond grains are attached on the steel particle.
Fig.1 SEM images of shot particles. (a) as-received (raw) steel particle, (b) carbon-black/steel (CBS) hybridized particle, and (c) diamond/steel (DS) hybridized particle.

Fig.2 Flowchart of specimen preparation.

Table 1 FPP condition.

<table>
<thead>
<tr>
<th></th>
<th>CBS particle</th>
<th>DS and raw steel particles</th>
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<tbody>
<tr>
<td>Peening apparatus</td>
<td>Air suction type, nozzle diameter φ6mm</td>
<td></td>
</tr>
<tr>
<td>Peening pressure</td>
<td>0.5 MPa</td>
<td></td>
</tr>
<tr>
<td>Nozzle distance</td>
<td>50 mm</td>
<td></td>
</tr>
<tr>
<td>Peening angle</td>
<td>90 deg.</td>
<td></td>
</tr>
<tr>
<td>Particle supply rate</td>
<td>5 g/s</td>
<td></td>
</tr>
<tr>
<td>Peening time</td>
<td>0.2 s</td>
<td>30 s</td>
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Then AISI304 stainless steel disk of 20mm in diameter and 5mm in thickness was treated with the double-FPP. Prior to the double-FPP, the disk was polished with emery papers. Two types of double-FPP specimens were prepared as shown in Fig.2. DS-CBS series were treated by FPP using the DS particle followed by that using the CBS particle. In contrast, CBS-DS series were treated by the inverse order. For comparison, specimens treated with FPP using the DS, CBS, or as-received (raw) steel particles and those without FPP treatment were also prepared (DS, CBS, S, and untreated series, respectively). Table 1 lists the FPP conditions using the CBS, DS and raw steel particles. After performing the double-FPP and FPP, the treated surfaces of the specimens were analysed using scanning electron microscope (SEM) and laser Raman spectrometer in order to discuss the transfer
phenomenon. Micro Vickers hardness was measured on the cross section the specimens. Surface roughness measurement was carried out with a contact profilometer. Tribological properties of the specimens were evaluated by conducting ball-on-disk reciprocating friction test. The mating material used was stainless steel ball of diameter 10mm. All tests were performed with a nominal load of 1.96N, a sliding speed of 200mm/min and a sliding stroke of 5mm without lubrication. Friction force was measured to obtain friction coefficient. After the tests, wear scars were analysed with an optical microscope (OM) to discuss the wear characteristics.

**Results and analysis**

Macroscopic observation of the specimens revealed that black colour was visible on the surfaces of the CBS and DS-CBS series, which were bombarded by the CBS particle. This indicates the presence of carbon-black on the surface due to transferring from the particle. However, the surface of the CBS-DS series did not show black colour clearly although it was treated with the CBS particle as the primary FPP sequence.

SEM observation and Raman spectroscopic analysis provide more details on the transfer phenomena. Fig.3 shows SEM images of the specimens observed with the back scattered electron detector. In these images, the dark contrast indicates the presence of light elements. It should be noted that millions of black dots are observed on the DS, CBS-DS and DS-CBS series. The size of the dots is basically similar to that of the diamond grains. Thus, the presence of the dots can be attributed to the diamond grains which were transferred from the DS particle and then embedded into the material. Raman spectra, shown in Fig.4, indicate the existence of diamond on the surface of the DS, CBS-DS and DS-CBS series, supporting the above discussion. For Raman spectra of the CBS and DS-CBS series, the peaks corresponding to $G$-band and $D$-band were observed. These were typical spectra obtained from carbon-black, thus the carbon-black transferred from the CBS particle exists on the surface of the CBS and DS-CBS series. The spectrum of the CBS-DS series only exhibits the peak corresponding to diamond without any trace of $D$-band and $G$-band peaks. This result means that carbon-black is no longer present on the CBS-DS surface, agreeing with the macroscopic observation.

![Fig.3 Typical back scattered electron images observed on the specimen surface.](image1)

![Fig.4 Raman spectra.](image2)
Results revealed that both of the carbon-black and diamond grains were present on the DS-CBS series surface while the CBS-DS series was only accompanied with diamond grains. For the CBS-DS series, carbon-black transferred during the primary FPP has been removed by the following FPP using the DS particle. The previous results implied that carbon-black transferred from the CBS particle adhere to the surface very weakly, probably due to Van der Waals force [5]. This nature allows the carbon-black to detach from the surface when the surface is treated with FPP. In contrast, the diamond grains transferred by the FPP using the DS particle were anchored to the material strongly, so that the grains transferred by the prior FPP step were maintained during the secondly FPP. This is the considerable reason why the surface of the DS-CBS series can be enriched with both of the carbon-black and diamond grains.

Fig.5 compares Vickers hardness of the specimens determined on the cross section. The double-FPP as well as the FPP increased surface hardness up to 500–600HV. Hardness of the CBS series was slightly lower than that of the other double-FPP and FPP specimens. This might be because of shorter peening time for the CBS series. Clear difference in hardness range was not observed among the DS, CBS-DS, DS-CBS and S series. This result is reasonable because the shot particles used for these specimens were basically identical except the presence or absence of the overlying carbon-black or diamond.

Table 2 lists the mean roughness $Ra$ and the maximum height $Rz$ measured on specimen surfaces. All series of the double-FPP and FPP specimens exhibited the almost similar roughness value: 0.7–1.0 $\mu$m in $Ra$ and 4–6 $\mu$m in $Rz$.

Fig.6 compares the friction coefficient of each specimen determined with the reciprocating ball-on-disk friction test. For the S and untreated series, the friction coefficient fluctuated severely around 0.4 before reaching stable level of approximately 0.6. No significant improvement in frictional characteristics was achieved by the FPP using the raw steel particle probably because this treatment did not enrich the surface with any carbon substances.

The DS and CBS-DS series showed similar variation in the friction coefficient; the value rose to 0.6 in the earlier cycles and then turned into slight decrease. These series are identical since both of them are modified with the embedded diamond grains. This is the reason for the similar feature in the
friction behaviour. The friction coefficient of these series obtained in the later stage of the test were lower than that of the untreated one. The authors have attributed this to powder-like wear debris produced from the surface, providing lubricative effects [6].

The DS-CBS and the CBS series, the specimens enriched with the transferred carbon-black, exhibited remarkably lower friction coefficient compared to other ones in the earlier cycles (up to 1000 cycles). This should be because the carbon-black acted as a solid lubricant. While the CBS series showed unexpected fluctuation in the friction coefficient in the later stage, it can be considered that the CBS and DS-CBS series has basically similar frictional characteristics.

As discussed above, transferred substances seemed to be a dominant factor to determine the value and variation in the friction coefficient. Focussing on the earlier stage of the test, the transferred carbon-black is effective to reduce the friction coefficient.

Fig. 6 Friction coefficient as a function of reciprocating cycles.

Fig. 7 Typical feature of wear scars after 2000 cycles of sliding.
To discuss the effect of the double-FPP and FPP on wear behaviour, the worn surfaces after 2000 cycles were observed with OM. Fig. 7 shows typical features of wear scars. Approximate width of the wear scars was determined from the OM observation and represented in this figure. Grooves are clearly observed on the worn surfaces of the untreated, S and CBS series. This feature implies severe scratching occurred on the contact area. Flattened regions which probably consist of eroded peaks and wear debris stacked around the peaks were observed on the wear tracks of the CBS-DS and DS series. In contrast, the DS-CBS series, enriched with both of the carbon-black and diamond grains, showed less damages on their wear track; no grooves appeared, and less plateaus were formed on the track. In addition, the width of the wear scar of the DS-CBS series was narrower than those of others. These results indicate the enhanced wear resistance of the DS-CBS series. It is supposed that the possible reason for this should be a synergic effect of the diamond grains and carbon-black concentrated on the surface. Double-FPP provides benefits to improve the tribological properties when it enriches two different substances on the one surface.

Conclusions
As a novel surface modification process for tribological application, the authors proposed double fine particle peening (double-FPP) using two types of hybridized particles: carbon-black/steel particle and diamond/steel one. The modified surfaces by using the double-FPP were evaluated with surface analyses and a tribological test. Conclusions resulted from this study are as follows:
(1) The double-FPP using the diamond/steel particle for the primary step and the carbon-black/steel one for the secondly step can create modified surface enriched with both of the diamond and carbon-black. In contrast, the double-FPP using the two particles with the inverse order only enriches the surface with the diamond.
(2) The double-FPP performed under the appropriate sequence reduces the wear as well as the friction in the early stage of sliding because of synergic effect of the transferred carbon-black and diamond. It has realized that the double-FPP has a potential to improve tribological properties of materials more remarkably than the conventional (single step) FPP.

References