PHASE IMAGING IN ATOMIC FORCE MICROSCOPY FOR CHARACTERIZING A WEAR SURFACE OF DLC COATING

H.-S. AHN
Tribology Research Center, Korea Institute of Science and Technology, 39-1, Hawolgok-dong, Songbuk-Gu, Seoul, 136-791, KOREA; e-mail: hsahn@kist.re.kr

S. A. CHIZHIK
Tribology Department, Metal Polymer Research Institute, 32A Kirov St, Gomel 246050, BELARUS; e-mail: schizhik@hotmail.com

SUMMARY
The phase contrast images in tapping mode atomic force microscopy was used to the characterization of inhomogeneity of thin tribofilm. Surfaces of diamond-like-carbon (DLC) coatings before and after friction contact against a steel ball slider were investigated. Comparison of the results obtained by atomic force microscopy with those of micro-Raman, AES and SIMS analyses and 3D-simulation of contact led to a speculation that the tribolfilms, composed almost of carbon element, may be graphite films or films mainly possessing graphitic property. The high contact pressure and temperature exerted to the real contact area can initiate the local transition of the diamond-like structure to the thin layers of graphite-like structure. This study demonstrated that inhomogeneity on worn surfaces can be effectively detected by phase imaging.

Keywords: atomic force microscopy, phase image, DLC coating, tribofilm, friction and wear

1 INTRODUCTION
The scanning probe microscopy technology allows studying mechanism of friction and wear on a micro- and nano-scale [1]. The efficiency of this method resides in that it provides complex analysis for characterizing a surface after friction contact. Atomic force microscopy (AFM) with oscillating mode of the cantilever is a powerful tool for the investigation of morphologies and mechanical properties of surfaces. In tapping mode (so-called intermittent contact) microscopy [2], a cantilever of force constant \( k_0 \) is forced to vibrate with amplitude \( A_0 \) at its resonance frequency \( \omega_0 \) in the absence of tip-sample interaction. Such a vibrating cantilever is then brought close to a sample surface so as to make it tap the surface with a set-point amplitude \( A_{sp} \) smaller than \( A_0 \). This amplitude is kept constant during scanning by the feedback mechanism. The phase shift of cantilever oscillation can be measured and used for phase imaging. Phase images contain important information related to micro-mechanical properties of the sample materials and adhesion generated between the probe tip and sample surface science they are highly sensitive to tip-sample force interaction [3–5]. Several works have reported the application of phase images for estimating structural heterogeneity of materials [2, 6–9].

Tribological behaviour of DLC coatings has been widely reported by numerous investigators. The wear mechanisms reported include formation of graphitic tribofilm on the contact surface of DLC coatings [10–12], degradation of mechanical properties (elastic modulus and hardness) without microstructural change in the bond structure of the DLC coating [13], material transfer from the film to the mating material or forming a layer of compact wear particles [14]. In the present work we demonstrated the significance of phase shift information of the atomic force microscopy on the evaluation of DLC coating worn surface at different wear stages.

2 EXPERIMENTAL DETAILS
The DLC coating used for this study was deposited using an unfiltered pulsed vacuum arc deposition system on the flat chromium-steel. The thickness of DLC coating was 1.4 \( \mu \)m as measured by the \( \alpha^{-} \)step profilometer (Tencor© P1 Long Scan Profiler). Dry oscillating wear tests were conducted at room temperature in a laboratory environment using a ball-on-plate tester. The balls were 3 mm diameter 52100 steel ball. The upper ball specimen was reciprocating with a velocity of 0.01 m/s against the stationary DLC-coated flat specimen for \( 2 \times 10^3 \) cycles. The applied load was 2.5 N.

Measurements of topography and phase shift were carried out using an AFM constructed in our collaborated laboratory. The oscillation of the probe was detected using an optical fiber interferometer. The probe was fabricated from a tungsten wire (100 \( \mu \)m diameter) by electrochemical etching. The following parameters were used for the tungsten probe installed in the AFM: \( R = 100 \) nm, \( \omega_0 = 44.9 \) KHz, \( Q_o = 166, \ k_o = 380 \) N/m, where \( R \) is tip radius, \( Q_o \) is quality factor of free cantilever. The mode of operation was tapping mode. The scanning set-point parameters employed were; \( r_{sp} = A_{sp}/A_0 = 0.5, \ A_0 = 20 – 80 \) nm. We used a stiff cantilever for characterizing the hard sample. This tapping system will be sensitive to the stiffness of the sample surface and the influence of adhesion will not be significant. Therefore, it is plausible to say that, in this investigation, the relative variation of stiffness of the
The experimental data showed a gradual decrease of friction coefficient until the test was terminated, indicating that the steady state was not achieved during the test period. After 2000 cycles, the wear scar diameter of the mating steel ball was about 620 \( \mu m \). The surface profile measurement of the wear track of the DLC coating using the \( \alpha \)-step profiler is shown in Fig. 1. It can be noted that the roughness decreased along the direction from the periphery to the center of the wear track where the surface was significantly smooth. The width of this smooth region was around 300 \( \mu m \), which was much smaller than the wear scar diameter formed on the steel ball slider.

The surfaces before and after the friction contact were studied with an AFM. As the surface morphology and the roughness of the worn surface were in general inhomogeneous, they might vary depending on the locations relative to the center of the wear track. The sample surface was divided into four zones, A, B, C and D, according to the distance from the center of the wear track (Fig. 1).

The topography and phase contrast image of the unworn surface (zone A, Fig. 2(a, b)) revealed that deposition technique used in this investigation produced elliptical clusters, with less than 800 nm in length. These DLC clusters formed individual asperities on the surface. The central zone of the wear track (zone D, Fig. 2(c,d)) were markedly different from the original surface. The clusters were not visible in the topography image (Fig. 2(c)) due to the surface films covering the coating surface. However, the phase contrast image (Fig. 2(d)) revealed the cluster structure underneath the films. There were many patches of relatively thick surface films deposited on the DLC clusters. It should be noted that considerable areas in the images were filled with very dark grey scale values and the rest with medium grey scale values. Both regions indicated the presence of materials with low stiffness (elastic modulus) but the dark grey regions exhibited relatively lower stiffness than the medium grey regions.

The images in Fig. 3 show the boundary region of the wear track (the zones B and C). The transition from the zones B to C was characterized by a sharp alteration in grey level from bright to dark grey scale values in the phase contrast image. The regions represented as bright grey scale values corresponded to the zone B, not the original surface (zone A), as confirmed by the size and shape of the clusters. The average size of the clusters in the zone B was about twice bigger than that in the zone A, and the asperities were comparatively flattened. The difference in the cluster morphology between the zones B and C was not noticeable. However, the phase contrast in the zone C was smaller than the zone B. The line profiles of topography and phase angle along the diagonal line, marked in the figures 3(a) and 3(b), revealed that the surface roughness and the phase angle significantly decreased in the zone C (Fig. 3(c)).

This roughness reduction was well correlated with the decrease of stiffness. The height difference between the films and the adjacent areas, that showed different grey scale values in the phase contrast image, was carefully measured using the topography information and was assumed as the thickness of the corresponding films. The approximate thickness of these films at various locations was in the range 5-17 nm. The dark grey sites were related to the regions of asperity contact and were attributed to the tribofilms that were less stiff than the initial DLC material: the darker the grey scale values, the greater the thickness of the tribofilm.
considerable smoothening of the surface occurred. Part of the wear track (zone D), one can notice that a roughness of the unworn surface (zone A) and central RMS phase shift deviation level ($\Phi$). The clusters in the worn surface became smaller and were not higher than 30 nm. Despite of the microgrooves, $R_q$ of the central part of the wear track was more than three times smaller than that of the unworn surface. The greater value of the parameter $\zeta$, after sliding contact, indicates that the surface became more isotropic. The smaller values of $\alpha_{\text{mid}}$ and $\alpha$ implied that the slope of asperities reduced, i.e., the asperities had a greater radius of curvature.

A 3-dimensional simulation of the real contact area was performed to visualize the contact pressure distribution at real contact areas [15, 16]. The darker regions corresponded to the regions with higher contact pressure, confirming the formation of the thicker tribofilms at the regions of maximum force interaction between the two surfaces.

Comparison of the results obtained by atomic force microscopy with those of micro-Raman, AES and SIMS analyses led to a speculation that the tribofilms, composed almost of carbon element, may be graphite films or films mainly possessing graphitic property. This is probably the main reason of the significant (more than three times) decrease of the friction coefficient observed in this work. The tribofilms formed on the worn surface protected the DLC coating surface inspite of their nanometer-scale thickness. The high contact pressure and temperature exerted to the contact surface can initiate the transition of the diamond-like structure to the graphite-like structure. The latter has lower elastic modulus than the former. Therefore, a significant decrease of the phase shift occurred when the probe tip traveled over these films. The films had the greatest thickness at the asperity summits where the local contact pressure was high and temperature flash was the most probable. It is likely that the transition from amorphous carbon to polycrystalline graphite dominantly occurred at the high asperity peaks.

### Table 1: Parameters for the roughness, tribofilm thickness and phase shift in the defined zones of the wear track on the DLC coating

<table>
<thead>
<tr>
<th>Zone</th>
<th>$R_q$, nm</th>
<th>$\alpha_{\text{mid}}$, deg</th>
<th>$\alpha$, deg</th>
<th>$S$, $\mu$m</th>
<th>$\zeta$,</th>
<th>$t$, nm</th>
<th>$\Delta\Phi_{\text{max}}$, deg</th>
<th>$\Phi$&gt;, deg</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>52.0</td>
<td>16.6</td>
<td>10.8</td>
<td>0.74</td>
<td>0.3</td>
<td>none</td>
<td>47.2</td>
<td>135.2</td>
</tr>
<tr>
<td>B</td>
<td>21.1</td>
<td>4.9</td>
<td>2.2</td>
<td>1.30</td>
<td>0.4</td>
<td>none</td>
<td>41.1</td>
<td>134.0</td>
</tr>
<tr>
<td>C</td>
<td>16.1</td>
<td>3.3</td>
<td>1.8</td>
<td>1.20</td>
<td>0.4</td>
<td>3-60</td>
<td>41.0</td>
<td>100.1</td>
</tr>
<tr>
<td>D</td>
<td>14.3</td>
<td>2.0</td>
<td>1.2</td>
<td>0.55</td>
<td>0.5</td>
<td>5-17</td>
<td>24.0</td>
<td>97.5</td>
</tr>
</tbody>
</table>

The clusters in the worn surface became smaller and were not higher than 30 nm. Despite of the microgrooves, $R_q$ of the central part of the wear track was more than three times smaller than that of the unworn surface. The greater value of the parameter $\zeta$, after sliding contact, indicates that the surface became more isotropic. The smaller values of $\alpha_{\text{mid}}$ and $\alpha$ implied that the slope of asperities reduced, i.e., the asperities had a greater radius of curvature.

4 CONCLUSIONS

This study demonstrated the significance of phase shift information of AFM on the evaluation of DLC coating worn surface. Study of the worn surface using the described technique revealed significant heterogeneity of the worn surface across the wear track. A tribofilm was formed at real contact regions and its thickness increased at the location experiencing higher contact pressure. Thin tribofilms having elastic modulus much lower then that of the DLC coating was formed on the DLC coating surface. Formation of these tribofilms may be related to the graphitization process of amorphous carbon by a tribochemical reaction.

The phase contrast imaging in atomic force microscopy showed a promise as an effective tool for better understanding of micromechanical properties of worn surfaces. It may provide insight into the formation of surface films and the influence of its microstructure on friction and wear process.

5 REFERENCES
