DLC Coatings Deposited by Magnetron Sputtering on metallic and Insulating Substrates

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Diamond Like Carbon (DLC)

Diamond-like Carbon (DLC) is a metastable form of amorphous carbon with significant sp3 bonding. DLC coatings are of interest in a wide variety of applications to provide low friction and high hardness and wear resistance.

Applications
- Glass: anti-scratch.
- Molds: prevent plastic transfer
- Engine parts: low friction (a-C).
- Space parts: low friction (a-C:H).
- Cutting tools: lubricated and dry conditions.
- Oil & gas: super lubricity in pipes.
- ...
**DLC: State of the art**

<table>
<thead>
<tr>
<th>Material</th>
<th>Preparation technique</th>
<th>Density, (gm/cm²)</th>
<th>% sp³</th>
<th>Hardness, (GPa)</th>
<th>Young’s modulus, (GPa)</th>
<th>Friction coefficient against metals*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diamond</td>
<td>Naturally occurring</td>
<td>3.52</td>
<td>100</td>
<td>100</td>
<td>1,050</td>
<td>0.02–0.10</td>
</tr>
<tr>
<td>DLC</td>
<td>Sputtering</td>
<td>1.9–2.4</td>
<td>2–5</td>
<td>11–24</td>
<td>140</td>
<td>0.20–1.20</td>
</tr>
<tr>
<td>Me doped H:DLC</td>
<td>Reactive sputtering</td>
<td>–</td>
<td>–</td>
<td>10–20</td>
<td>100–200</td>
<td>0.10–0.20</td>
</tr>
<tr>
<td>H:DLC</td>
<td>r.f plasma</td>
<td>1.57–1.69</td>
<td>–</td>
<td>16–40</td>
<td>145</td>
<td>0.02–0.47</td>
</tr>
<tr>
<td>H:DLC, DLC</td>
<td>Ion beam</td>
<td>1.8–3.5</td>
<td>–</td>
<td>32–75</td>
<td>–</td>
<td>0.06–0.19</td>
</tr>
<tr>
<td>ta-DLC</td>
<td>Vacuum arc</td>
<td>2.8–3.0</td>
<td>85–95</td>
<td>40–100</td>
<td>500</td>
<td>0.04–0.14</td>
</tr>
<tr>
<td>ta-DLC</td>
<td>PLD</td>
<td>2.4</td>
<td>70–95</td>
<td>30–60</td>
<td>200–500</td>
<td>0.03–0.12</td>
</tr>
</tbody>
</table>

Ion assistance required ~ 100eV


DLC hard coating magnetron deposition

Anode to extend the plasma

Magnetically linked magnetrons

Prof. Wolf-Dieter Munz “Industrial scale deposition of well adherent and low friction DLC coatings grown by HIPIMS and anode assisted unbalanced magnetron” – Private discussion
DLC hard coating magnetron deposition

55GPa Microhardness, comparable to filtered arc

Prof. Wolf-Dieter Munz “Industrial scale deposition of well adherent and low friction DLC coatings grown by HIPIMS and anode assisted unbalanced magnetron” – Private discussion
Ion assisted deposition on insulators?

Magnetron magnetic design (electron filter) + modified asymmetric bipolar pulsed PS

- Positive ion bombardment
- No electron bombardment: Reduced thermal load?

Proprietary technology: Patent application number GB1605162.5 (March 2016)
**Ion assisted deposition on insulators?**

Magnetron size 400x100mm² (industrial relevant) – WC, Graphite targets
HIPIMS Ti/Cr + WC improves coating adhesion
Optimized magnetic field configuration to enhance ion assisted deposition

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Ion assisted deposition on insulators?

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HIPIMS Ti/Cr + WC improves coating adhesion
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The distribution of ion energies at the substrate in an asymmetric bi-polar pulsed DC magnetron discharge

3 energy regimes are measured
Coating Hardness: above 30 GPa on floating substrate

Berkovich indenter
Single indents: 2, 7, 12 mN
Load-partial Unload: 12 mN
Hysitron TI950

Hardness = 32 Gpa
Young modulus = 220 Gpa
500nm thick DLC
DLC hard coating magnetron deposition

15GPa Microhardness with -75V Bias!

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Ion assisted deposition on insulators?

Hardness > 30GPa  Hardness < 12GPa

Modified DC-Pulsed PS  Commercial DC-Pulsed PS

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Metallic substrates: HIPIMS implantation to promote coating adhesion
DLC

HSS Compliant layer

M2 HSS

Epitaxial growth

a-C:W

Compliant layer
Adhesion on metals: Standard Rockwell (Daimler or Mercedes) tests

DLC on HSS

- DLC delaminated coating
- HSS substrate surface
- DLC coated surface

Indentation

Cr + WC + DLC on HSS

- NO DLC coating delamination

HF5

Rockwell HRC tests - 150kg - Diamond indenter conical tip
Friction and wear – ASTM G99 Standard test

HF1

DLC coated SS304

SS304 uncoated substrate
Friction and wear – ASTM G99 Standard test

![Friction coefficient vs. time graph]

Friction coefficient vs. time graph showing the change in friction coefficient over time (t) from 0 to 8000 seconds. The graph displays a steady decrease in friction coefficient with time.
Low accumulated stress @ high hardness

Biased process: high DLC coating stress

N4E process: low DLC coating stress

Ongoing stress measurements in DLC coatings
Insulating substrates: High hardness DLC for abrasion resistance
DLC coating on Glass

Delamination problem: Need to reduced thicknesses and ion bombardment

Hardness in the range of 20GPa for 40nm thick DLC layer
DLC coating on Glass

Substrate can be pre-treated with reactive gases such as oxygen

Current to floating potential (10mV/A)
DLC coating on Glass: substrate pre-treatment

Untreated glass substrate
RMS roughness: 2.6 nm

Oxygen treated glass substrate
RMS roughness: 1.4 nm
### DLC coating on Glass: Taber abrasion tests

<table>
<thead>
<tr>
<th>Coating description</th>
<th>TABER Test (7.5N)</th>
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<tbody>
<tr>
<td>DLC 1: 30 min O2 plasma cleaning + 6nm DLC-a</td>
<td>≤20 times NG</td>
</tr>
<tr>
<td>DLC 3: 15 min O2 plasma cleaning + 6nm DLC-a</td>
<td>≤20 times NG</td>
</tr>
<tr>
<td>DLC 2: 30 min O2 plasma cleaning + 3nm DLC-a</td>
<td>≤20 times NG</td>
</tr>
<tr>
<td>DLC 6: 5 min O2 plasma cleaning + 3nm DLC-a</td>
<td>≤10 times NG</td>
</tr>
<tr>
<td>DLC 4: 15 min O2 plasma cleaning + 6nm DLC-b</td>
<td>≤20 times NG</td>
</tr>
<tr>
<td>DLC 7: 5 min O2 plasma cleaning + 6nm DLC-b</td>
<td>≤15 times NG</td>
</tr>
</tbody>
</table>

Substrate plasma pre-treatment improved DLC coating adhesion
DLC coating on Glass: nano-scratch tests

500mN critical load – 5 micron tip radius
Thank you for your attention!