Plasma Processes as a Cleaner Alternative for Cleaning, Corrosion Resistance, and Functionalization of Metallic Surfaces

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Those who are inspired by a model other than Nature, a mistress above all masters, are laboring in vain. - Leonardo Da Vinci
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PROGRAMA DE PÓS GRADUAÇÃO

ÁREA DE CONCENTRAÇÃO DE PESQUISA

CÊNCIA E TECNOLOGIA DE MATERIAIS
OBJECTIVES

✓ Development of clean and efficient high vacuum technologies
✓ Replacement for traditional methods for metallic or polymeric surfaces
✓ Treatments to clean, deposit thin films, and functionalize surfaces
✓ One-dimensional nanotechnology
✓ Development of environmentally friendly and sustainable technologies
TREATMENT SEQUENCE

- Removal of oil from aluminium surfaces
- Deposition of a thin film of HMDSO
- Functionalization of the HMDSO film
PLASMA EQUIPMENT *

Fabricated by Diener Electronic - Plasma Surface Technology, Germany, Series Pico with a cylindrical chamber with capacity of 5 liters.

The generator works with radio-frequency signal of 40 kHz and maximum power of 200W.

Pump Ilmvac mark, with 1.8 m³/h and pressure of 3 × 10⁻² mbar

* Made available by SABO
VARIABLES STUDIED

• Gas composition

• Reactor Power

• Pressure

• Exposure Time
CLEANING PROCESS

• Radio-frequency signal of 40 kHz, maximum power of 200W

• Pressure of 0.075 Torr

• Oxygen (99% of purity) immediately introduced.

• Contact angle – Before treatment, 90°; After, 12.6°
DEPOSITION PROCESS

- Monomer: HDMSO, co-reagent: Argon
- HMDSO pressure: 0.03 mbar; Ar pressure: 0.18 mbar
- Power: 30, 45, 60W; time: 5-30 min.
- Most effective conditions - pressure: HMDSO 0.03 Torr; Ar pressure: 0.15 Torr; Power: 30W; time: 15 minutes
- Thickness of the layer obtained: 943 mm; deposition rate: 62.9 mm/min

Note: Film Functionalization in Progress
XPS RESULTS-CLEANING

✓ Reduction of the concentration of atomic C 1s from 86.14 to 44.47% due to the removal of the protective oil from the surface of aluminum,

✓ Increase in the atomic concentration of O 1s from 12.55 to 27.97%.

✓ Increase in the concentration of atomic Al 2p from 1.34 % to 27.55%.
<table>
<thead>
<tr>
<th></th>
<th>Contact Angle</th>
<th>Surface Free Energy (mN/m)</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>$\gamma^d_L$ (mN/m)</td>
</tr>
<tr>
<td>Pure water</td>
<td></td>
<td>95.5</td>
</tr>
<tr>
<td>Ethylene glycol</td>
<td></td>
<td>77.0</td>
</tr>
<tr>
<td>Before treatment</td>
<td>plasma</td>
<td>7.6</td>
</tr>
<tr>
<td>After treatment</td>
<td>Plasma</td>
<td>76.1</td>
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SALT SPRAY TESTS

EVALUATION OF CORROSION RESISTANCE ALUMINUM SAMPLES PROTECTED BY THE HDMSO FILMS

ASTM B 117, ASTM D 1654, E ABNT NBR 8094

TEST CONDITIONS USED WERE: 5% ± 1% AQUEOUS SOLUTION OF SODIUM CHLORIDE (THE PH OF THE SOLUTION WAS ADJUSTED FROM 6.5 TO 7.2). THE AIR TEMPERATURE WAS MAINTAINED AT 35 ± 2 °C

RESULTS

CONSIDERABLE IMPROVEMENT: PROTECTED PLATES CORRODED ONLY WHERE PROTECTIVE FILM DID NOT COAT UNIFORMILY- EVEN AFTER 3 WEEKS EXPOSURE
CONCLUSÕES

• Significant surface modification was obtained with plasma exposure time of 30 seconds.

• Using argon plasma, after the activation step the contact angle obtained was 12.6°, associated to an increase in surface energy of 95.48 mN/m;

• The argon plasma doesn’t introduces new chemical groups in the surfaces. But is effective in surface cleaning, and surface crosslinking. However we observe the introduction oh —oh groups, when the sample is exposed to air, after the argon plasma treatment.

• The plasma process is clean, with very low production of residues, consisting in a ecologically friendly technique.

• Using plasma technology is possible develop an industrial process to clean, deposit and activate surfaces with very low ecological damage compared to wet processes.
MUITO OBRIGADO