hip-V HiPIMS for the coating industry

Dr. Iván Fernández Martínez

Shenzhen, June 16th, 2018
Outline of the presentation

1.- Company presentation.
2.- Some aspects of HiPIMS technology.
3.- Industrial implementation of hip-V
4.- hip-V HiPIMS power supplies.
Plasma sputtering
Coating solutions
Dr. Iván Fernández Martínez, co-founder, managing director of Nano4Energy SL and hipV AB. Physicist, over 16 years in sputtering and particularly, in HiPIMS.

Ambiorn Wennberg, co-founder and business manager of Nano4Energy SL and hipV AB. Material Science Engineer. His background is in metallurgy, vacuum technology.

Prof. Fernando Briones, co-founder of Nano4Energy SL. Material Science Engineer. Over 45 years experience in R&D. Over 300 published papers.

Alvaro Mendez, degree on Chemistry. PhD on HiPIMS with EU Marie Curie Action.

Sandra Muñoz Piña, degree on Chemistry. PhD on sputtering coatings with GLAD.
What do we offer?

Core technology: Magnetron sputtering.

Plasma process development (magnetron, process controller, etc ...)

- Gencoa (UK) as strategic partner

High power electronics / HiPIMS power supplies

- Viesca (ES) as strategic partners

Characterization of thin film properties

- Access to european research center facilities.
# History of the hip-V power supplies

<table>
<thead>
<tr>
<th>Year</th>
<th>Event</th>
</tr>
</thead>
<tbody>
<tr>
<td>2008</td>
<td>1st proof of concept</td>
</tr>
<tr>
<td>2012</td>
<td>First industrial 6kW prototype (Gen 1)</td>
</tr>
<tr>
<td>2014</td>
<td>Generation 2 1st commercial version</td>
</tr>
<tr>
<td>2016</td>
<td>Generation 3 Made for the industry</td>
</tr>
</tbody>
</table>

Complete plasma operation functionality provided by Dr. Fernandez in 2007 and manufactured by Ingeniería Viesca SL.
Flexibilidad
Nos adaptamos a las necesidades específicas del cliente, con diseño a medida para cada proyecto.

Equipo de tracción para tranvía histórico (1913) en Soller (Mallorca)
Who manufactures hip-V HiPIMS?

<table>
<thead>
<tr>
<th>Year</th>
<th>Certification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>2008</td>
<td>Certified as a qualified supplier of KNORR-BREMSE</td>
<td></td>
</tr>
<tr>
<td>2009</td>
<td>Certified as a qualified supplier of ALSTOM</td>
<td></td>
</tr>
<tr>
<td>2009</td>
<td>Certified as a qualified supplier of CAF Power</td>
<td></td>
</tr>
<tr>
<td>2010</td>
<td>ISO 9001: 2008</td>
<td></td>
</tr>
<tr>
<td>2011</td>
<td>ISO 14001: 2004 certification in progress</td>
<td></td>
</tr>
<tr>
<td>2014</td>
<td>Certified as a qualified supplier of TALGO</td>
<td></td>
</tr>
<tr>
<td>2015</td>
<td>ISO 9100: 2009</td>
<td></td>
</tr>
</tbody>
</table>
Viesca > 5000 units worldwide

**REFERENCE LIST**

<table>
<thead>
<tr>
<th>END USER</th>
<th>DIRECT CLIENT</th>
<th>EQUIPMENT</th>
<th>QTY</th>
<th>SUPPLY DATE</th>
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<tbody>
<tr>
<td>THERMAL POWER PLANT ANDORRA (Teruel - ESPAÑA)</td>
<td>G.E. - ENERGY</td>
<td>STATIC SWITCH</td>
<td>67</td>
<td>2005-2009</td>
</tr>
<tr>
<td>ELECTROPLATING</td>
<td>EGALSA</td>
<td>15kW 400Vac / 10V-1500A CONVERTER</td>
<td>160</td>
<td>2005-2009</td>
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<tr>
<td>TBA BUENOS AIRES</td>
<td>MERAK</td>
<td>28 kVA 750Vac / 400V 50Hz INVERTER</td>
<td>342</td>
<td>2008-2011</td>
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<tr>
<td>ARGEL TRAM</td>
<td>MERAK</td>
<td>28kVA 750Vac / 400V 50Hz INVERTER</td>
<td>157</td>
<td>2008-2009</td>
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<tr>
<td>MOSCOW METRO</td>
<td>MERAK</td>
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<td>943</td>
<td>2009-2014</td>
</tr>
<tr>
<td>VITORIA LRV</td>
<td>MERAK</td>
<td>38 kVA 750Vac / 400Vac 50Hz INVERTER</td>
<td>26</td>
<td>2008</td>
</tr>
</tbody>
</table>

**LIGHT RAIL VEHICLES LOCATION 55kVA APS**

<table>
<thead>
<tr>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>BUDAPEST</td>
</tr>
<tr>
<td>FRIBURGO</td>
</tr>
<tr>
<td>TALLIN</td>
</tr>
<tr>
<td>TAIWAN</td>
</tr>
<tr>
<td>CASIARI</td>
</tr>
<tr>
<td>CINCINNATI</td>
</tr>
<tr>
<td>SIDNEY</td>
</tr>
<tr>
<td>ESTOCOLMO</td>
</tr>
<tr>
<td>BESANCON</td>
</tr>
<tr>
<td>BIRMINGHAM</td>
</tr>
<tr>
<td>CUIABA</td>
</tr>
<tr>
<td>DEBRECEN</td>
</tr>
<tr>
<td>CADIZ</td>
</tr>
<tr>
<td>NANTES</td>
</tr>
<tr>
<td>GRANADA</td>
</tr>
<tr>
<td>MALAGA</td>
</tr>
<tr>
<td>SEVILLA</td>
</tr>
<tr>
<td>ZARAGOZA</td>
</tr>
<tr>
<td>VITORIA</td>
</tr>
<tr>
<td>HELSINKI</td>
</tr>
<tr>
<td>CAMBERRA</td>
</tr>
<tr>
<td>XINTIAN</td>
</tr>
<tr>
<td>KANSAS</td>
</tr>
</tbody>
</table>

www.ingenieriaviesca.com

kW/MW power supplies for 24/7 operation in railway sector mainly.
Frank Papa, founder and president of GP Plasma, LLC. He represents Nano4Energy in the USA and China. He has worked at Hauzer Techno Coating, Crystallume Inc., Vergason Technology and Gencoa Ltd. His background is in sputtering, arc and CVD processes and hardware development. He has developed the HiPIMS technology at Hauzer.

Feng Limin / 冯利民, 工程热物理专业；豪泽技术涂层5年销售经理；上海新弧源涂层技术有限公司6年总经理； represents Nano4Energy in China.国内独家代理.

Gerhard Eichenhofer, founder and president of 4APlasma (Germany). Sales manager at hipV AB. He has worked at Solvix, ENI and Advanced Energy. His background is in sputtering, arc and CVD processes and hardware development.
**What do we do?**

**Product Overview:**

<table>
<thead>
<tr>
<th></th>
<th>hiPV 1KW</th>
<th>hiPV 6KW</th>
<th>hiPV 10KW</th>
<th>hiPV 10KW</th>
<th>hiPV 20KW</th>
<th>hiPV 20KW</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Power</strong></td>
<td>1KW</td>
<td>6KW</td>
<td>10KW</td>
<td>10KW</td>
<td>20KW</td>
<td>20KW</td>
</tr>
<tr>
<td><strong>Peak Power</strong></td>
<td>0.1MW</td>
<td>0.5MW</td>
<td>1.2MW</td>
<td>1.2MW</td>
<td>2.4MW</td>
<td>2.4MW</td>
</tr>
<tr>
<td><strong>Voltage</strong></td>
<td>1200V</td>
<td>1200V</td>
<td>1200V</td>
<td>1200V</td>
<td>1200V</td>
<td>1200V</td>
</tr>
<tr>
<td><strong>Current max. HiPIMS</strong></td>
<td>100A</td>
<td>500A 1000A option</td>
<td>1000A</td>
<td>2000A</td>
<td>2000A</td>
<td>1000A</td>
</tr>
<tr>
<td><strong>Current DC max.</strong></td>
<td>3A</td>
<td>18A</td>
<td>25A</td>
<td>25A</td>
<td>50A</td>
<td>50A</td>
</tr>
<tr>
<td><strong>Frequency max.</strong></td>
<td>40KHz</td>
<td>2KHz P_{max} @1KHz</td>
<td>2KHz P_{max} @1KHz</td>
<td>1KHz</td>
<td>1KHz</td>
<td>2KHz P_{max} @1KHz</td>
</tr>
<tr>
<td><strong>Time ON</strong></td>
<td>5-1000μs</td>
<td>5-1000μs</td>
<td>5-1000μs</td>
<td>5-1000μs</td>
<td>5-1000μs</td>
<td>5-1000μs</td>
</tr>
<tr>
<td><strong>ARC Control</strong></td>
<td>&lt; 3μs</td>
<td>&lt; 3μs</td>
<td>&lt; 3μs</td>
<td>&lt; 3μs</td>
<td>&lt; 3μs</td>
<td>&lt; 3μs</td>
</tr>
<tr>
<td><strong>Cooling</strong></td>
<td>Air</td>
<td></td>
<td></td>
<td>Water &amp; Air</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Biggest market for hipV*
hip-V 10kW hiPIMS unit (mostly used)

- Allows DC, HiPIMS and Bias operation (units are fully exchangeable!!).
- Already in production for large coating system builders worldwide.

Output Data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Output-Power</td>
<td>10kW</td>
</tr>
<tr>
<td>Output Voltage</td>
<td>0V to -1000V, optional 1200V</td>
</tr>
<tr>
<td>Output Current</td>
<td>1000A (pulse peak) max. 25A DC, for &lt;400V in DC-mode</td>
</tr>
<tr>
<td>Pulse frequency</td>
<td>1kHz at 1000/1200V, 1000A, with lower energy pulses the frequency can be increased</td>
</tr>
<tr>
<td>Regulation</td>
<td>Voltage / Current / Power</td>
</tr>
<tr>
<td>Pulse width</td>
<td>5μs to 300 μs; 1000μs (optional) or DC</td>
</tr>
<tr>
<td>Duty cycle</td>
<td>&lt;50% or DC 100%</td>
</tr>
<tr>
<td>Arc detection / handling</td>
<td>&lt;3μs</td>
</tr>
<tr>
<td>Arc level current / voltage</td>
<td>Adjustable 50A ÷ 1500A, Voltage delta in %</td>
</tr>
<tr>
<td>Voltage stability</td>
<td>±2.5%</td>
</tr>
<tr>
<td>Voltage ripple</td>
<td>&lt;5%rms</td>
</tr>
</tbody>
</table>
**HiPIMS in industrial production: Key aspects for successful operation (**)**

1. **Reliable operation** in production is a must (24/7 operation).

2. Plasma **process understanding** by the **manufacturer**. It is a complex process!!.

3. Demonstrate it is possible to achieve **deposition rates** comparable to DC sputtering... Advanced solutions such as anodes, Positive V reversal, etc... are required.

4. **PLC communication** with the latest protocols (Profibus, Ethercat, ......).

5. Ability to run **stable in reactive sputtering**.

6. Ability to run **stable Metal Ion etching**. Widely used process to improve adhesion in arc systems.

(**) Based on our customers feedback
Facilities

Vacuum chambers (x3 different sizes)
Magnetrons:
- Circular 2"
- Rectangular 20x7.5cm²
- Gencoa rectangular 40x10cm²

Gases: Ar, O2, N2, He, H, D....
SpeedFlo reactive process controller.
Power supplies (up-to-date):
- AC Dual Magnetron 40kHz (AE/10kW)
- DC-Pulsed 150kHz (EN Tech/10kW)
- 3x hipV HIPIMS + Bias (6-20kW)
- RF
**What do we offer?**

**Core technology**: Magnetron sputtering.

- Plasma process development (magnetron, process controller, etc ...)
  - Gencoa (UK) as strategic partner

- High power electronics / HiPIMS power supplies
  - Viesca (ES) as strategic partners

- Characterization of thin film properties
  - Access to european research center facilities.
What do we offer?

Strong UBM: key for hard coatings!!
Products & process implementation for V±EE DLC
Hugo Huang (黄汉乐)
hugo@gencoaa.com

Lane Bao (鲍磊 先生)
Shanghai Triplewins
Lane.bao@www-vacuum.com
Outline of the presentation

1. Company presentation.

2. Our history with HiPIMS.

3. Some aspects of HiPIMS.

4. Industrial implementation of hip-V

5. Summary
• Higher plasma density → Higher ionization of gases and sputtered material.
• Higher ion assistance to the growing film → Denser and smoother films.
First experiments with HiPIMS in lab. scale magnetrons... 2008!

Evidences of highly ionized tungsten metal plasma
Simulation of Effects of Ion/Neutral Ratio

Is HiPIMS the solution for everything?

..... NO!!! (But there are a few industrial nitches such as smooth hard coatings where neither magnetron sputtering or cathodic arc can beat HiPIMS...).
Some technical aspects of HiPIMS that make them very very beneficial for hard coatings:

Bias synchronizing, allows:

1. Selectivity on the element for the metal ion assistance.
2. Reduced Ar incorporation: Coating stress release.
3. Metal ion etching and implantation.
Single phase metastable cubic $\text{Ti}_{1-x}\text{Al}_x\text{N}$ ($x<0.64$) with low stress ($<0.7$ GPa).

Ti+ irradiation gives rise to two phase cubic $\text{TiAlN} + \text{hexagonal AlN}$ (brittle, 17 GPa).

Reduced damage with low energy assistance

G. Greczynski et al, SCT 206, 4202 (2012)
1.- Selection of metal ions with Bias sync.

HiPIMS/DC Magnetron 1

Bias HiPIMS

Sync to Ti$^+$ assistance (Magnetron 2)

Floated at Magnetron 1 operation

HiPIMS Magnetron 2
Bias pulse is sync and extended to collect the highest amount of ions (bias current) with low risk of arcing.
Benefit of Bias synchronizing:

2.- Reduced Argon incorporation: Coating stress release
2. Coating stress release: Bias sync.

- Magnetron Discharge (metal ion generator)
- Sample Bias (Ion assistance)


Thick coatings!!
1. - Selection of metal ions with Bias sync.

G. Greczynski et al, JVST A 32, 041515 (2014)
H. Fager et al, JAP 121, 171902 (2017)

 alloys without external heating (T_s ≤ 150°C). Using substrate bias synchronized with the Ta⁺/Ta²⁺ portion of the HIPIMS-pulses provides effective near-surface atomic mixing resulting in film densification. The substrate potential V_s = V_f at all other time, which minimizes Ar⁺ bombardment that typically leads to high compressive stress. DCMS sputtering from the Ti and
I bias = 500A
I target = 1500A
V target = 1000V
V Bias = 500V
I bias = 500A
I target = 1500A
15us delay
Benefit of High Voltage Sync. Bias:

3.- Allows metal ion etching and implantation
3. - Metal ion etching

Biased component

Ti plasma

Sample Bias (Ion assistance)

Magnetron Discharge (metal ion generation)

Graphs showing:
- Voltage (kV) vs. Time (us)
- Sample Bias (V) vs. Time (us)

Diagrams showing species:
- Ti⁺
- Ti²⁺
- Ti⁴⁺
3. Metal ion etching and implantation

3. Metal ion etching and implantation

**Cr (1A/cm²)**

- Cr (1+)
- Cr (0)
- Cr (2+)

**Ti (3A/cm²)**

- Ti (1+)
- Ti (1+)/Ar(1+)
- Ti (2+)

**Cr**: Higher population of neutrals (not affected by Bias)

**Ti**: Metal etching with low bias values → reduces arcing issues!!

3. - Metal ion etching and implantation

\[ E_i = E_0 + Qe \left( V_{\text{plasma}} - V_{\text{surface}} \right) \]

Cr\(^{+1}\), Cr\(^{+2}\), Ti\(^{+1}\), Ti\(^{+4}\)

Effective etching

Net deposition (no etching)
Local epitaxy between HSS substrate and Ti bonding layer at RT
**Compositional measurements: EELS**

**Titanium on HSS:**
Implantation depth: 6 nm

**Chromium on HSS:**
Implantation depth: 7 nm

I. Fernandez-Martinez et al, **accepted** in SCT (doi:10.1016/j.surfcoat.2018.04.090)
Critical loads above 100N!!

Example of H-free DLC coated parts
Conclusion 1:

The proper Bias operation with HiPIMS allows smooth coatings with good adhesion and low stress levels. (Not achievable with Magnetron DC or Cathodic Arc)
Deposition rates in HiPIMS:

4.- Need advanced strategies such as the presence of anodes, HiPIMS V+,...
The implementation of magnetically guided anodes (or AC Dual) can boost the film ion incorporation.

Hip-V is a floated HiPIMS power supply!!
HiPIMS with positive V reversal

- New N4E development. Patent application number GB1713385.1
HiPIMS with positive V reversal

2. Demonstrated >25% increase in deposition rate for Me-N and ta-C in different industrial machines (for example in PVT and Tekniker batch coaters).
3. Generate enhanced coating ion assistance $\rightarrow$ denser coating structure.
4. Reduced arc appearance in reactive sputtering.

<table>
<thead>
<tr>
<th>Dep. rate [$\mu$m/hr]</th>
<th>0.51</th>
<th>0.42</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness [GPa]</td>
<td>21</td>
<td>17</td>
</tr>
<tr>
<td>Positive Pulsing</td>
<td>YES</td>
<td>NO</td>
</tr>
</tbody>
</table>

25% increase in ion incorporation!!
(deposition rate and hardness)
HiPIMS V+ for ta-C: 35 GPa Hardness

Hardness = 36 GPa
Young’s Modulus = 248 GPa

More energetic ions → higher sp³ hybridization

Triboindenter TI950 from Hysitron equipped with a diamond Berkovich indenter.
High energy ions generated in the switching electric field.
**Hip-V for glass Al metallizing**

Centurion System from Duralar Technologies
Table diameter = 950 mm
*Rotatable Magnetrons – 152 mm Diameter*
1200 mm Length
Average Power to Cathode = 5kW
Coating thickness approximately 1μm
Room Temperature
Work done in cooperation with Von Ardenne North America

Courtesy of Duralar Technologies
Hip-V for glass Al metallizing

Reflectivity improvement with high $I_{peak}$ & $V+$
hip-V in reactive sputtering
Process stabilization is more complex than standard DC or Pulsed-DC.
Long experience in reactive control process using Gencoa - SpeedFlo

Regulation modes: $I_{pk}$, V or Optical monitoring. Proven technology!!
**HIPIMS Ti-based coatings in production**

**Ti (400mm²) Ar + Nitrogen**
3kW 120us – 500Hz $I_{\text{peak}}$: 220 – 500A peak

**Nitrogen flow at setpoint**
- 45% ~ 4.5 sccm
- 35% ~ 5.5 sccm
- 25% ~ 7.0 sccm
HiPIMS in reactive sputtering

$I_{\text{peak}}$ control Al-O : 5hrs deposition run

14us – 3kHz 450V
Ar flow 150sccm
High tendency to arc at the beginning

$I_{pk}$ regulation mode for the most complex process : Al in Ar + Oxygen!!
Core technology: Magnetron sputtering.

What do we offer?

Plasma process development (magnetron, process controller, etc ...)

- Gencoa (UK) as strategic partner

High power electronics / HiPIMS power supplies

- Viesca (ES) as strategic partners

Characterization of thin film properties

- Access to European research center facilities.
Characterization facilities for ta-C on tools

**ta-C on microtools. Al machining!!**

Interferential colour: ta-C

Raman in microtools for sp2/sp3 ratio in sharp edges
Industrial implementation of N4E technology in industrial OEM’s:
Industrial HIPIMS 80kW average

3 meters coating machine

4 x 20 kW power module (HIPIMS + Bias)
PVT introduces its first DLC-coating system based on the most advanced process technologies. In cooperation with Nano4Energy SL (N4E), Madrid, Spain, the coating system xPro4C was designed and developed using N4E’s HiPIMS V+ (positive pulse) proprietary technology. This magnetron sputtering mode, in combination with the Variable magnetics (VT-ER) provided by Gencoa Ltd., allows an accurate control of both the flux and energy of the ion species bombarding the substrate. And this is the crucial mechanism to produce coatings with optimum properties such as crystallinity, density, smoothness or internal stress.
Hip-V for smooth Me-N and PECVD

3-fold rotation
2 x magnetrons
5kW average each
-60V bias
650 mm diameter x
650 mm height

23-25GPa
250 C

1μm/hr!! for CrN and ZrN
Thank you for your attention!