Engineering Coating Structure by Suspension Plasma Spray

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Concordia University

Thermal Spray of Suspensions and Solutions (TS4)
Montréal, Canada, 2-3 December 2015
Outlook

• Introduction
• Coating buildup in SPS
• Diagnostics of In-flight particles
• Tailoring coating characteristics and properties
  • Hydrogen production
  • Super-hydrophobic coatings
• Conclusion
Faculty of Engineering and Computer Science (ENCS)
ENCS FAST FACTS:

UNDERGRADUATE STUDENTS

3894

UNDERGRADUATE PROGRAMS: 9
DEPARTMENTS: 6
FULL TIME FACULTY MEMBERS: 176
PROFESSIONAL ENGINEERS: 95%

GRADUATE STUDENTS

3267

CANADA RESEARCH CHAIRS: 5
INDUSTRIAL RESEARCH CHAIRS: 4
CONCORDIA RESEARCH CHAIRS: 21
Concordia Institute of Aerospace Design and Innovation - CIADI

• Mission
  – Promote and support research activities through a coordinated university-wide platform
  – Operate an aerospace internship program for graduate and undergraduate students
Research Themes

- Modelling and Simulation
- Micro Sensors and Materials
- Avionics and Electrical
- Environment, Social, Economic Impact
- Energy and Power
- Structures, Materials and Manufacturing
- Space and Autonomous Systems
Suspension Thermal Spraying (STS)

- Fine droplets formed by injection of submicron powders (STS) in the flame
- Cooling rates exceeding 10 million °C/sec → formation of nanograin

Grain size range (20-80nm)
Challenges – Research Opportunities

- **Liquid Feedstock**
  - Stable high-power plasma torch
  - Understanding liquid processing
  - Understanding coating formation

- **Plasma/Liquid Processing**
  - Proper and stable injection of the liquid feedstock
  - Stable suspensions and solutions
  - Suspension particle size?
  - Storage - Handling of nanoparticles

- **Coating Architecture**
  - Heat flux
  - Local mass flux to substrate

- Development or adaptation of measurement techniques and sensors for droplets and fine particles

A. Vardelle, C. Moreau, 4th International Round Table on Thermal Plasmas, Marrakesh, 2014
Suspension/Jet Interaction

- Complex phenomena occur sequentially:
  - Primary and secondary atomization
  - Liquid evaporation
  - Sintering and melting of the spray material

Reduced Particle Impact Velocity

- Critical to control the microstructure

![Particle Velocities](image)

- Highly sensitive to particle size and material density
- Higher free stream velocities can dramatically increase the impact velocity of small particles

J. Oberste-Berghaus, et al., ITSC Proceedings 2005, Basel (Switzerland)
Coating Build-up

- STS typical microstructure formation depends strongly on the particle velocity and diameter at impact:
  - Normal direction
  - Parallel direction

\[
St = \frac{\rho_p d_p^2 v_p}{\mu_g l_{BL}}
\]

Deflection of the particle trajectory depends on the Stokes number \( St \):

Controlling SPS Coating Structure

- A designed cooling system allows:
  - driving out the low-velocity particles before their impact onto the substrate
  - maintaining the nominal temperature of the substrate by playing on the cooling gas flow rate

A. Joulia, et al., JTST, 24 (2014) 24-29
In-flight Particle Diagnostics

- Key parameters influencing the coating structure:
  - Particle temperature, velocity (module and direction) and diameter
  - Substrate temperature and substrate

- Challenges
  - Small particle diameter
  - High number density of particles
Light Diffraction Method

Laser

Plasma torch

Test section

Incident light

Lens

Scattered light

Smaller particle

Intensity signal

angle

0

20
Characterization of In-flight Particles in Plasma Spray by Light Diffraction

Original measurement

Plasma effect on signal

Wide range of influential wavelengths

Raw signal in receiver

Corrected measurement

Acceptable plasma effect in signal

Mechanical shielding

Selected wavelength

Optical filtering

Measurement signal

Signal filtering
Particle Size Distribution of In-flight Particles in Plasma Spray

Original measurement

Corrected measurement

Particle Diameter (μm)

Cumulative volume (%)

Volume frequency (%)
Validation of Measurement Result

Off-line Measurement

On-line Measurement

Cumulative volume (%) vs. Particle Diameter (μm)

Volume frequency (%) vs. Particle Diameter (μm)
Thermal and Non-thermal Radiation

- Plasma Radiation
- Thermal radiation emitted by the in-flight particles
- Plasma radiation scattered by the in-flight particles
- Radiation from plasma surrounding the in-flight particles
Front Measurement

- ND Filters for attenuation.
- Long pass filters (600 nm).
- The particle’s average trajectory was aligned to the torch centerline using Acuraspray.
- The aim is to assess the power density incident on the particles
Results of Front Spectra at 80 cm

- Plasma only.
- Plasma + Ethanol.
- Plasma + Ethanol + Powder.
Effect of the Scattered Radiation

- 1st window set 995 / 785 nm.
- 2nd window set 995 / 877 nm.
Spectra Collected Radially

- Plasma only.
- Plasma + Ethanol.
- Plasma + Ethanol + Powder.
Amorphous Content in SPS Alumina / Zirconia Coatings: Role of Melting Time

SPS: Amorphous Formation in In-flight Agglomerated Nano-particles

Different degrees of melting and homogenization

Alkaline Water Electrolysis

\[ \text{H}_2\text{O} = \text{H}_2 + \frac{1}{2} \text{O}_2 \]

Cathode: \[ 2\text{H}_2\text{O} + 2 \text{e}^- = \text{H}_2 + 2 \text{OH}^- \]

Anode: \[ 2 \text{OH}^- = \frac{1}{2} \text{O}_2 + \text{H}_2\text{O} + 2 \text{e}^- \]

Material selection:
Alkaline Water Electrolysis

\[ \text{H}_2\text{O} = \text{H}_2 + \frac{1}{2} \text{O}_2 \]

Cathode: \[ 2\text{H}_2\text{O} + 2 \text{e}^- = \text{H}_2 + 2 \text{OH}^- \]

Anode: \[ 2 \text{OH}^- = \frac{1}{2} \text{O}_2 + \text{H}_2\text{O} + 2 \text{e}^- \]
Objectives

To improve the energy efficiency of the alkaline water electrolysis process for hydrogen production using thermal spraying for manufacturing nickel cathode electrodes
## Spray Parameters

<table>
<thead>
<tr>
<th>Process</th>
<th>Standoff distance (mm)</th>
<th>Feeding rate (g/min)</th>
<th>Plasma gas flow (Ar/H₂) (SLPM)</th>
<th>Torch traverse speed (m/s)</th>
<th>Current (A)</th>
<th>Number of passes</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS</td>
<td>80</td>
<td>15</td>
<td>60/2</td>
<td>0.5</td>
<td>500</td>
<td>8</td>
</tr>
<tr>
<td>SPS</td>
<td>60</td>
<td>31</td>
<td>50/3</td>
<td>0.5</td>
<td>450</td>
<td>20/6</td>
</tr>
</tbody>
</table>

Combined atmospheric and suspension plasma spray processes to take advantage of multiscale surface structure.
Top Surface and Cross-section

AES

Substrate temperature: 90 to 150°C
Particle temperature: 2126±213°C
Particle velocity: 101±31 m/s

Substrate temperature:
200 to 220°C in first pass
390 to 450°C in last pass
Top Surface and Cross-section
Electrocatalytic Activity

Working electrode: Workpiece
Counter electrode: Pt
Reference electrode: Hg/HgO
Electrocatalytic Active Electrode Coatings for Hydrogen Evolution

- Electrodes with both nano and micron-scale microstructures have a larger surface area where:
  - larger micropores promote the electrolyte diffusion and facilitate H₂ bubble ascension from the pores
  - nanosized features provide more active sites.

Problem: In-flight Icing

• Presence of super-cooled micro-droplets
• Icing accumulation on aerodynamic surfaces

Drag ↑  Lift ↓  Efficient engine power ↓
Anti-icing Superhydrophobic Coatings

- Water drops bounce back before freezing
- Heated de-icing is facilitated
Background: Wettability

Hydrophilic (WCA < 90°)

Hydrophobic (WCA > 90°)

Superhydrophobic

Bhushan & Jung (2011)

Eigenbrod et al. (2011)

150° < WCA
Effect of Roughness on Wettability

- Flat substrate
- Nano-structured
- Micro-structured
- Hierarchical structure

Air pocket

Lotus Leaf

Bhushan & Jung (2011)
Motivation

Superhydrophobicity

Low surface energy

- Mainly polymeric and organic
- Low durability
- Poor mechanical properties

Surface morphology

- Complex and costly
- Difficult to apply on large-scale surfaces
APS Tests

Low velocity / high temperature

• Contact Angle = 128.2° ± 2.6
• Sliding Angle > 60 °

High velocity / Low temperature

• Contact Angle = 145.4° ± 2.4
• Sliding Angle > 60 °

• Generally low mobility
SPS – Suspension in Water

- Completely dispersed
- Very stable
SPS – Suspension in Water

- Forms aggregated
- Relatively low stability
SPS Results

Suspension in water

- Contact Angle = 156° ± 3
- Sliding Angle = 9.8° ± 4.8

Suspension in ethanol

- Contact Angle = 167° ± 3
- Sliding Angle = 1.3° ± 0.3

SPS in water

SPS in ethanol
Comparison of Mobility

- **WX2100**: an off the shelf polymeric spray

<table>
<thead>
<tr>
<th>Surface</th>
<th>Coefficient of Restitution (impact velocity = 250 mm/s)</th>
<th>Coefficient of Restitution (impact velocity = 450 mm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPS-W</td>
<td>0.82</td>
<td>0.38</td>
</tr>
<tr>
<td>SPS-E</td>
<td>0.82</td>
<td>0.48</td>
</tr>
<tr>
<td>WX2100 (commercial spray)</td>
<td>0.73</td>
<td>0.48</td>
</tr>
<tr>
<td>Lotus leaf</td>
<td>0.75</td>
<td>0.40</td>
</tr>
<tr>
<td>Nano-grass</td>
<td>0.83</td>
<td>0.45</td>
</tr>
</tbody>
</table>
Conclusion

• The microstructure of suspension thermal spray coatings presents unique characteristics that can be tailored for specific applications

• Numerous challenges (research opportunities) must be addressed to improve our understanding of SPS processes and improve their reproducibility
Thanks