Thermal Spray Coatings
For Industrial Applications

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Coatings for severe operating environment

Heavy combination of mechanical stresses and aggressive environment

Temperature

Wear

Corrosion

Mechanical stress

Oxidation

Wear

Corrosion

Oxidation

Temperature
Coatings for severe operating environment

R&D plays a key role for component working in critical operating conditions

Aim of presentation:
Description of case studies related to innovations and R&D in thermal spray field
Surface engineering by thermal spray process

A thermal spray process can be divided into four sections:

- **heat/energy source** (thermal energy for heating and melting)
- **material feed/flow**
- **material spray** (kinetic energy for propelling dispersion)
- **material deposition**
Surface engineering by thermal spray processes

Thermal spray processes

Combustion
  - Low velocity
  - High velocity
    - Flame-wire
    - D-Gun®
    - Flame-powder
      - HVOF
      - HVAF

Cold spray
  - Plasma
    - Air
    - Chamber
      - APS
      - VPS–LPPS
      - CAPS

Electrical
  - Electric arc
    - Arc spraying
Surface engineering by thermal spray processes
Coating formation

- Void
- Oxidized particle
- Unmelted particle
- Roughened substrate
- Coating
- Environment
- Substrate
- Powder

From flame:
- $\text{O}_2$, $\text{N}_2$, $\text{CO}$, $\text{H}_2$

$\text{H}_2\text{O}$, oxide, etc.
• An inert gas is passed through a potential differential.
• An arc is formed between the two dipoles.
• The gas is ionized and recombines after going through a free expansion.
• The recombination effect releases heat and creates a plasma flame ("plume").
• Powder is inserted into the flame and is propelled towards the substrate.
Plasma spraying

- Water cooled cathode (tungsten)
- Powder injector (external)
- Plasma flame = plume
- Plasma jet
- Arc and plasma (5 – 10 % of the gases are ionized.)
- Water cooled nozzle
- Plasma gases (primary and secondary)
  - Primary gases: Ar, N₂
  - Secondary gases: H₂, He
- Current (amperage is set)
  Voltage range: 50 – 500V
Plasma spraying

- Max. plume temperature $\approx 12000 \, ^\circ \text{C}$
- Impact speed of particles $200-400 \, \text{m/s}$
- In-flight time of particles $\approx 10^{-3} \, \text{s}$
- Heating/cooling rate $\approx 10^7 \, \text{K/s}$
Plasma spraying

Process parameters

- Plasma (Primary) gas flow
- Secondary gas flow
- Plasma power
- Spray distance
- Injector angle, length and size
- Carrier gas flow
- Gun spray angle
Primary gases (Ar, N$_2$): plasma stability and transport properties

Secondary lighter gases (He, H$_2$), enthalpy adjustment.

N$_2$ and H$_2$ are diatomic gases. These plasmas have higher energy contents for a given temperature than argon and helium because of the energy associated with dissociation of molecules.
Plasma spraying

Spray distance

- Powder particle temperature vs. distance from the nozzle
  - Small particles, low powder density
  - Big particles, high powder density

- Flow temperature vs. distance from the nozzle

- Flow velocity vs. distance from the nozzle

- Particle velocity vs. distance from the nozzle
  - \( \text{Ar/H}_2 \)
  - \( \text{N}_2/\text{H}_2 \)
Plasma spraying

Spray distance

Particle velocity

Distance from the nozzle

29 kW
20 kW
(same gas comp.)

Oxide content

Surface roughness

Cold particles
Plasma spraying

**Controlled Atmosphere Plasma Spray**

Chamber is backfilled after evacuation, either at

- near vacuum (1 - 10 mbar) **VPS**
- reduced pressure (> 50 mbar) **LPPS**
- standard pressure **APS**
- elevated pressure (up to 4 bar) **HPPS**
- with a substitute atmosphere (reactive (CxHy), inert (Ar)) **RPS / IPS**

**VPS / LPPS / LVPS**

- spraying in near vacuum or under reduced pressure conditions
- spray particles are unimpeded by frictional forces of the atmosphere
Plasma spraying

Materials

- Ceramics (high melting point)
  - $\text{Al}_2\text{O}_3$, $\text{Al}_2\text{O}_3/\text{TiO}_2$, $\text{Cr}_2\text{O}_3$, $\text{ZrO}_2$ - $\text{Y}_2\text{O}_3$, $\text{La}_2\text{Zr}_2\text{O}_7$, ecc.
  - $\text{ZrB}_2$, $\text{TiB}_2$, $\text{SiC}$ (non standard)

- Metals (refractory, oxidation control is mandatory, controlled atmosphere)
  - MCrAlY, Ni-and Mo-based alloys

- Cermets
  - $\text{Cr}_3\text{C}_2$ - based (high T, generally NiCr matrix)
  - $\text{WC}$ - based (better mechanical properties, $T_{\text{max}}$ 500 °C (decarburation), Co - CoNiCr - based matrix)
Plasma spraying

Cermet $\text{Cr}_3\text{C}_2$ – Ni-Cr

- Low porosity
- Lamellar microstructure, partially molten ceramic phase
Coating formation – plasma vs. HVOF

Plasma spraying → hot particles

HVOF spraying → fast particles

thermal energy

flame seems tighter because particles are colder and shine less

kinetic energy
HVOF deposition

- Max. jet temperature \( \approx 2800 \, ^\circ \text{C} \)
- Particles impact speed \( 400-700 \, \text{m/s} \)
- In-flight time \( \approx 10^{-3} \, \text{s} \)
- Heating/cooling rate \( \approx 10^7 \, \text{K/s} \)

Plasma Spray

HVOF
Applications

Wear and corrosion resistant coatings
(Mechanical, chemical, aerospace, textile industries)
- Cermet (WC, Cr$_3$C$_2$ or TiC in Co, Co-Cr or Ni-Cr metal matrix);
- Ceramics (Cr$_2$O$_3$, Al$_2$O$_3$-TiO$_2$);
- Metallic (Ni or Co base, e.g. NiCrBSiC).

Thermal barrier coatings (TBC)
(Aerospace, energy industries)
- ZrO$_2$ - Y$_2$O$_3$.

High temperature oxidation resistant coatings
(Aerospace, energy, chemical industries)
- Ni - Cr – Al base (e.g. NiCoCrAlY).

Biocompatible coatings
Porous Ti, hydroxy apatite (Ca$_{10}$(PO$_4$)$_6$(OH)$_2$)
Materials and Surface Engineering Lab

Research partners and clients

- AIRBUS DEFENCE & SPACE
- GE Oil & Gas Nuovo Pignone spa
- WÄRTSILÄ
- Agenzia Spaziale Italiana
- ThalesAlenia Space
- ANSV
- Avio Aero
- esa
- MBDA Missile Systems
- aviospace
- Avio
Aircraft engines and power gas turbines

Max gas temperature in turbine: 900 - 1400 °C

Rise in gas temperature in turbine

↓

Improvement of thermodynamic efficiency
Turbine blades protection

High temperature and combustion products (e.g. SO$_2$, SO$_3$, V$_2$O$_5$) cause surface damages on the Ni-base superalloy.

- **Hot corrosion** (700 – 925 °C)
  (SO$_3$ attack, sulphides formation)

- **High temperature oxidation**
  (T > 1000° C)

- **Erosion**
  (Solid particles in gas stream)

**Max service temperature of superalloy!**

**Protective ceramic overlay**

**Thermal Barrier Coating (TBC)**
Thermal Barrier Coatings (TBC)

Top Coat (ZrO$_2$–6-8%Y$_2$O$_3$)

TGO (a-Al$_2$O$_3$)

Bond Coat (Ni/Co-CrAlY)

Substrate (Ni–base superalloy)

- Thermal insulation
- Erosion protection

ZrO$_2$ permeable to O$_2$ (through open porosity & by ionic conduction)

- Oxidation barrier
- Enhanced adhesion
- Hot corrosion resistance
- Thermo-mechanical loading

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Innovative materials for gas turbines

Strategies to improve turbines performance: use of CMC materials
Innovative materials for gas turbines

Strategies to improve turbines performance:
use of CMC materials

Modification of substrate-coating system

<table>
<thead>
<tr>
<th>Patent No.</th>
<th>Date of Patent</th>
<th>References Cited</th>
</tr>
</thead>
<tbody>
<tr>
<td>4,642,271</td>
<td>2/1987</td>
<td>Rice</td>
</tr>
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<td>5,026,604</td>
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</tr>
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<td>5,407,740</td>
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</tr>
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<td>5,427,986</td>
<td>6/1995</td>
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<tr>
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<td>1/1997</td>
<td>Moore et al.</td>
</tr>
<tr>
<td>5,817,432</td>
<td>10/1998</td>
<td>Chwastiak et al.</td>
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</table>
Innovative materials for gas turbines

Strategies to improve turbines performance: use of CMC materials

Modification of substrate-coating system

GE Rolls-Royce – development of F136 engine (second choice for F-35 aircraft)
3rd low pressure stage – stator in SiC/SiC
Innovative materials for gas turbines

Strategies to improve turbines performance: use of CMC materials

Modification of substrate-coating system

GE Successfully Tests World’s First Rotating Ceramic Matrix Composite Material for Next-Gen Combat Engine

F414 low-pressure turbine blades prove silicon carbide CMC material for unprecedented deployment in GE’s adaptive cycle combat engine

February 10, 2015

CINCINNATI, OH – February 10, 2015 – GE Aviation successfully tested the world’s first non-static set of light-weight, ceramic matrix composite (CMC) parts by running rotating low-pressure turbine blades in a F414 turbofan demonstrator engine designed to further validate the heat-resistant material for high-stress operation in GE’s next-generation Adaptive Engine Technology Demonstrator (AETD) program currently in development with the United States Air Force Research Lab (AFRL).
Innovative materials for gas turbines

Strategies to improve turbines performance: use of CMC materials

Modification of substrate-coating system

Problem:
- Active oxidation of SiC/SiC, especially in presence of H₂O steam: *Thermal & Environmental Barrier Coatings*
Innovative materials for gas turbines

Environmental Barrier Coatings

**EBC deposited by APS (examples)**
- \( \text{HfO}_2 - \text{Y}_2\text{O}_3 - \text{Gd}_2\text{O}_3 - \text{Yb}_2\text{O}_3 \)
- Barium-Strontium-Alumino-Silicate (BSAS)
Need for innovation in surface engineering
Case study: Hard chrome replacement

Hard Chrome plating is an electrolytic method of depositing chrome for engineering applications, from a chromic acid solution.

**Cr\(^{6+}\) compounds**

**EU classification**
COMMISSION REGULATION (EU) No 348/2013
of 17 April 2013


<table>
<thead>
<tr>
<th>Substance</th>
<th>Intrinsic property(ies) referred to in Article 57</th>
<th>Transitional arrangements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromium trioxide</td>
<td>Carcinogenic (category 1A) Mutagenic (category 1B)</td>
<td>21 September 2017</td>
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<tr>
<td>EC No: 215-607-8 CAS No: 1333-82-0</td>
<td></td>
<td></td>
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<tr>
<td>Acids generated from chromium trioxide and their oligomers</td>
<td>Carcinogenic (category 1B)</td>
<td>21 September 2017</td>
</tr>
<tr>
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This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 17 April 2013.

For the Commission
The President
José Manuel BARROSO

Dept. of Chemical and Materials Engineering (ICMA) - Sapienza University of Rome
Need for innovation in surface engineering

Hard chrome replacement

Properties of hard chrome:

- LOW COEFFICIENT OF FRICTION – coefficient against steel of 0.16 (0.21 dry),

- HIGH HARDNESS
  Typical values of 850 - 1050 HV

- WEAR RESISTANCE
  extremely good resistance to abrasive and erosive wear

- CORROSION RESISTANCE
  extremely high resistance to atmospheric oxidation, and a good resistance to most oxidising and reducing agents

- MACHINING
  Can be successfully finished
Case study

Hard chrome replacement in marine engine components

Wärtsilä aims to be the leader in power solutions for the global marine markets
Case study

Hard chrome replacement in marine diesel engine components
Case study

Hard chrome replacement in marine diesel engine components
Case study

Hard chrome replacement in marine diesel engine components

Valves problems:

- High temperature
- Wear
- Cold and hot corrosion

Hot-corrosion and oxidation

Thermo-mechanical fatigue

Wear and cold corrosion from sulphur compounds

Creep

Fatigue

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Case study

Hard chrome replacement in marine diesel engine VALVES

Problem:
Wear and cold corrosion in exhaust and intake valves stem

Hard chrome on stem has a short service-life
Case study

Hard chrome replacement in marine diesel engine valves

Ceramic-metal (cermet) HVOF coatings

2 phases - microstructure

Hard ceramic phase: wear resistance

Metal matrix: toughness and corrosion resistance
Case study

Hard chrome replacement in marine diesel engine valves

Materials (Sulzer powders):

✓ WC-CoCrNi  WCN
✓ (WC-Co)-NiCrSiFeBC  WSF
✓ Cr₃C₂ – 25 (Ni-Cr)  CRC

Facility:

HVOF – JP5000 gun
Wärtsilä requirements and constrain

Cermet HVOF coatings

- Thickness higher than 150 µm
- Porosity as low as possible
- Hardness higher than 900 HV
- High deposition efficiency

Optimization of deposition parameters by
*Design of Experiment (DoE)*

**2 factors** | **3 levels** | **3² parameter sets**
--- | --- | ---
1) Kerosene - O₂ flow | 1) Low | Keros. (gph) – O₂ (scfh)
2) Spray distance | 2) Medium | 5.5-1700
 | 3) High | 6-1850
 |  | 6.5-2000
| Distance (mm) |
| 320 |
| 355 |
| 380 |
Phase 1- HVOF coatings optimization (DoE)

Investigated properties

- Deposition efficiency
- Porosity as a function of total flow and spray distance
- Hardness

Weighted combination of these properties provide the “desirability function”
## WC-CoCrNi

### Properties

<table>
<thead>
<tr>
<th>Properties</th>
<th>Optimized value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deposition efficiency (um/pass)</td>
<td>24,3</td>
</tr>
<tr>
<td>Hardness (HV$_{100}$)</td>
<td>1610</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>3,04</td>
</tr>
<tr>
<td><strong>Desirability</strong></td>
<td><strong>0,84</strong></td>
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</tbody>
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### Keros. (gph) – O$_2$ (scfh)

<table>
<thead>
<tr>
<th>Distance (mm)</th>
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<tr>
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</table>

Optimized parameters

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Dept. of Chemical and Materials Engineering (ICMA) - Sapienza University of Rome
(WC-Co) - NiCrSiFeBC

<table>
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<th>Properties</th>
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</thead>
<tbody>
<tr>
<td>Deposition efficiency (µm/pass)</td>
<td>10,8</td>
</tr>
<tr>
<td>Hardness (HV&lt;sub&gt;50&lt;/sub&gt;)</td>
<td>1053,5</td>
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<tr>
<td>Porosity (%)</td>
<td>1,79</td>
</tr>
<tr>
<td>Desirability</td>
<td>0,82</td>
</tr>
</tbody>
</table>

**Properties Optimized value**

- **Deposition efficiency (µm/pass)**: 10,8
- **Hardness (HV<sub>50</sub>)**: 1053,5
- **Porosity (%)**: 1,79
- **Desirability**: 0,82

**Keros. (gph) – O<sub>2</sub> (scfh)**

<table>
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<th>6,5-2000</th>
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<tr>
<td>400</td>
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<tr>
<td>370</td>
<td></td>
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<td></td>
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<tr>
<td>340</td>
<td></td>
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</tbody>
</table>

Optimized parameters
Cr$_3$C$_2$ – 25 (Ni-Cr)

### Properties

<table>
<thead>
<tr>
<th></th>
<th>Optimized value</th>
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<tbody>
<tr>
<td>Deposition efficiency</td>
<td>34,3</td>
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<tr>
<td>(um/pass)</td>
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<tr>
<td>Hardness (HV$_{100}$)</td>
<td>1369</td>
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<tr>
<td>Porosity (%)</td>
<td>4,06</td>
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<td>Desirability</td>
<td>0,996</td>
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</table>

### Optimized parameters

<table>
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<th>Distance (mm)</th>
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</table>

Diaグラフ O2 mass flow
Y: Desirability

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Phase 1 - HVOF coatings optimization (DoE)

Optimized coatings

WCN

CRC

WSF

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Phase 2- optimized coatings characterization

Corrosion tests

Wear tests
Phase 2- optimized coatings characterization

Corrosion tests

1 hour immersion test – boiling 5% H₂SO₄ solution

<table>
<thead>
<tr>
<th>Mass loss (mg/cm²)</th>
<th>CRC</th>
<th>WSF</th>
<th>WCN</th>
<th>HC</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>8.29</td>
<td>6.26</td>
<td>0.065</td>
<td>191.02</td>
</tr>
</tbody>
</table>

**Corrosion test - mass lost**
Phase 2- corrosion tests

Hard chrome

Not protective
Phase 2- corrosion tests

WCN coatings

No surface damage detectable
Phase 2- optimized coatings characterization

Wear tests

- Load: 91 N
- Sliding distance: 2000 m
- Speed: 1 m/s

![Image of wear test results](image_url)

- As sprayed
- Post corrosion attack

- CRC_STL: 0.42
- CRC_NSA: 0.49
- WSF_STL: 0.23
- WSF_NSA: 0.27
- WSN_STL: 0.14
- WSN_NSA: 0.16
- WCN_STL: 0.22
- WCN_NSA: 0.13
- HC_STL: 0.02
- HC_NSA: 0.01

h.c. — not available
Phase 3 - coating selection for valve deposition

**WC-CoCrNi:**
- Highest hardness
- Lowest wear rate
- Lowest corrosion rate
- Good density
- High deposition efficiency

- WC-CoCrNi coating was selected
- Coated valves tested for 3 years on a Wartsila test engine
Innovative coatings for hot corrosion

Case study: Hot corrosion on marine diesel engines valves

Aggressive environment produced by high impurity contents within the fuel: V, Na, S.
Innovative coatings for hot corrosion

Multiphase cermet coatings: CrystalCoat

<table>
<thead>
<tr>
<th>(19) United States</th>
</tr>
</thead>
<tbody>
<tr>
<td>(12) Patent Application Publication</td>
</tr>
<tr>
<td>VERLOTSKI</td>
</tr>
<tr>
<td>(10) Pub. No.: US 2008/0317966 A1</td>
</tr>
<tr>
<td>(43) Pub. Date: Dec. 25, 2008</td>
</tr>
</tbody>
</table>

| (54) | THERMALLY SPRAYED GASTIGHT PROTECTIVE LAYER FOR METAL SUBSTRATES |
| (30) | Foreign Application Priority Data |
| | Jun. 19, 2007 (DE) 102007028109.0 |

| (75) Inventor: Vadim VERLOTSKI, Wuppertal (DE) |
| Correspondence Address: KATTEN MUCHIN ROSENMAN LLP 575 MADISON AVENUE NEW YORK, NY 10022-2585 (US) |

| (73) Assignee: MARKISCHES WERK GMBH, Halver (DE) |
| Int. Cl. |
| B05D 1/10 (2006.01) |
| B05D 1/00 (2006.01) |
| B32B 5/00 (2006.01) |
| H01L 21/316 (2006.01) |

| (52) U.S. Cl. 427/452; 427/576; 428/220; 106/287.18 |
| ABSTRACT |
In a thermally sprayed, gastight protective layer for metal substrates, especially those based on Fe, Ni, Al, Mg and/or Ti, wherein the spray powder for the purpose comprises at least
Innovative coatings for hot corrosion

Multiphase cermet coatings: CrystalCoat

- Metal alloy
- SiO$_2$
- Basalt
- Oxide
- Other silicates

CRYSTALCOAT

Mineral-Metal Coating

Substrate
Innovative coatings for hot corrosion

Case study: Hot corrosion on marine diesel engines valves

Aim: development of innovative thermal spray coatings alternative to Crystal Coat

Commercial (non standard) tested solutions

- $\text{Cr}_3\text{C}_2 - \text{NiCr}$
- $\text{Cr}_3\text{C}_2 - \text{NiCrAlY}$
- $\text{Cr}_3\text{C}_2 - \text{CoNiCrAlY}$
- $\text{Cr}_3\text{C}_2$ – self fusing alloy

Development of innovative solutions

- Mullite – nano $\text{SiO}_2 - \text{NiCr}$
Innovative coatings for hot corrosion

Phase 1- coatings optimization (DoE)

- Thickness higher than 150 µm
- Porosity as low as possible
- High deposition efficiency

Optimization of deposition parameters by
Design of Experiment (DoE)

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</table>
Example: optimization of Cr$_3$C$_2$ - self fusing alloy

Optimized coating

Ceramic phase dispersed in multiphase metallic matrix

Very low porosity (< 1 %)
Innovative coatings for hot corrosion

✓ Mullite – nano SiO₂ – NiCr

Powder agglomeration – spray drying

Nano-SiO₂ cover completely the spray dried agglomerates
Innovative coatings for hot corrosion

✓ Mullite – nano SiO$_2$ – NiCr

Coating deposition

Coatings retain the nano-structure
Innovative coatings for hot corrosion

Hot corrosion tests

- Temperature: 750 °C
- Environment: Na$_2$SO$_4$ – V$_2$O$_5$ molten salt
- Exposure time: up to 100 h

Oxide scale evolution
Innovative coatings for hot corrosion

Hot corrosion tests

Nanostructured coating
Innovative coatings for hot corrosion

Hot corrosion tests

Comparison between nanostructured and self fusing coatings

**Cr3C2 – self fusing alloy**

![Nano 25 h](image)

**Nano 25 h**

Thinner oxide scale in nano-coating
Corrosion kinetics

Scale growth kinetics

- Carbide-NiCr
- Carbide-MCrAlY/2
- Carbide-Self flux
- Carbide-MCrAlY/1
- Cer-nano/oxide-NiCr

Thickness [μm] vs Time [h]