Mesoporous beta-Silicon Carbide (β-SiC) and Carbon materials for innovative solutions
1. Company profile
   1.1. Business area
   1.2. Historical background
   1.3. Team

2. Key properties of β-SiC

3. Current standard SiC products

4. Capabilities for SiC customization

5. Newly developed mesoC+™ Carbon support
1. COMPANY PROFILE:
1.1. BUSINESS AREA (1/2)

Self-bonded porous β-Silicon Carbide

- Strong chemical resistance
- High thermal conductivity
- Outstanding mechanical strength
- Strong thermal and hydrothermal resistance
- High surface area and controlled porosity

BRINGS EXTENSIVE BENEFITS IN HARSH CONDITIONS
Fischer-Tropsch, Selective hydrogenation, Biomass conversion…
1. COMPANY PROFILE:
1.1. BUSINESS AREA (2/2)

SiC customization to fit customers' specific requirements

Surface area
from 10 to 130 m²/g

Shape
from powders to foams

Purity
3 purity grades

Porosity
pore size distribution and pore volume

Surface chemistry
coatings, composites, doping

WHAT ARE YOUR NEEDS? LET'S TALK!
2001 SICAT is created and funded with US$4 million by investors and CNRS (French National Research Agency)
Exclusive patent license on beta-SiC materials with CNRS
Small pilot facility in Alsace near Strasbourg

2006-09 Joint development with oil industry leader

2010-2011 Opening of ACM’s plant in Willstätt, Germany.
Industrial feasibility demonstrated on a 40 Mt/yr batch facility

2012-2014 Joint development on GTL
EU-sponsored FREECATS program and French Agency-sponsored OPTISOL program
French Agency-sponsored COPHOTOFE program

2015 First commercial order
Two projects at end stage on selective hydrogenations
EU-sponsored PRINTCR3DIT program

2016 First delivery of High-Purity SiC pellets (15.5 Mt)

2017 Launch of mesoC+™: mesoporous carbon pellets with outstanding mechanical strength
Highly experienced scientists and professionals

Managing director:
  Jean-Bernard Lartigue (former CEO - TOTAL Petrochemicals)

Finance, administration, legal:
  Guillaume Bougard

Research and development:
  Dr. Charlotte Pham
  Dr. Nelly Batal
  Angélique Becker

Technical development:
  Christophe Vieville

Scientific Advisor:
  Dr. Jan Lerou (former CTO - Oxford Catalysts)
1. Company profile

2. Key properties of β-SiC
   2.1. General
   2.2. Porosity
   2.3. Surface chemistry
   2.4. Robustness: mechanical and chemical
   2.5. Flexibility of the production process

3. Current standard products

4. Capabilities for customization

5. Newly developed mesoC+™ Carbon support
2. KEY PROPERTIES OF β-SiC:

2.1. GENERAL

**SICAT’s β-SiC**

- Self-bonded material, no binder, not sintered
- Medium surface area, meso-macro porous network
- Thin layer of $\text{SiO}_x\text{C}_y$ on surface

**Conventional α-SiC**

- High mechanical strength
- Chemically inert, resistant to acids, bases, sulfation and oxidation
- High thermal stability and conductivity
2. KEY PROPERTIES OF β-SiC:

2.2. POROSITY

Typical values

*Water pore volume of 0.4 cc/g*

*Packed density: 800 g/l*

Typical values from Hg intrusion

*Meso and macroporosity*

*Total pore volume of 0.5 cc/g*

Typical values from N$_2$ isotherm

*BET SA: 25 m$^2$/g*

*No micropores (< 5 m$^2$/g)*

*Pore volume: 0.2 cc/g*
Amorphous $\text{SiO}_2/\text{SiO}_x\text{C}_y$ on surface allowing for an easy active phase deposition

HRTEM image of a sample showing an amorphous layer over the surface

XPS spectra from Si-2p

After TT 700°C-air

Raw $\beta$-SiC
2. KEY PROPERTIES OF β-SiC: 2.4. ROBUSTNESS (1/2)

Grain Crushing Strength

<table>
<thead>
<tr>
<th>Shape</th>
<th>Crushing Strength*</th>
<th>Bed Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pellets: Ø 3 mm (Pore Volume: 0.5 cc/g)</td>
<td>&gt; 40 N/mm</td>
<td>800 g/l</td>
</tr>
<tr>
<td>Rings: Ø 8/5 mm (Pore Volume: 0.5 cc/g)</td>
<td>&gt; 10 N/mm</td>
<td>500 g/l</td>
</tr>
<tr>
<td>Spheres: Ø 5.5 mm (Pore Volume: 0.5 cc/g)</td>
<td>260 N</td>
<td>800 g/l</td>
</tr>
<tr>
<td>Open cell foams</td>
<td>&gt; 2 MPa</td>
<td>200 g/l</td>
</tr>
</tbody>
</table>

* Grain method per ASTM 4179 and 6175

Attrition loss

<table>
<thead>
<tr>
<th>Shape</th>
<th>Fines*</th>
<th>Bed Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pellets: Ø 3 mm (Pore Volume: 0.5 cc/g)</td>
<td>&lt;1%</td>
<td>800 g/l</td>
</tr>
</tbody>
</table>

* per ASTM D4058-87
2. KEY PROPERTIES OF $\beta$-SiC:  
2.4. ROBUSTNESS (2/2)

Chemical stability

<table>
<thead>
<tr>
<th></th>
<th>Fresh $\beta$-SiC</th>
<th>HCl 37% 2 weeks</th>
<th>HNO$_3$ 70% 2 weeks</th>
<th>NaOH 10M 2 weeks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight change</td>
<td>-</td>
<td>- 0.3%</td>
<td>- 0.2%</td>
<td>- 1.3%</td>
</tr>
<tr>
<td>Crushing strength</td>
<td>82 N/mm</td>
<td>86 N/mm</td>
<td>82 N/mm</td>
<td>91 N/mm</td>
</tr>
<tr>
<td>BET Surface Area</td>
<td>25 m$^2$/g</td>
<td>23 m$^2$/g</td>
<td>25 m$^2$/g</td>
<td>24 m$^2$/g</td>
</tr>
</tbody>
</table>

Resistance under 45 bar of steam at 255°C

<table>
<thead>
<tr>
<th></th>
<th>Fresh $\beta$-SiC</th>
<th>3 weeks after</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight change</td>
<td>-</td>
<td>- 0.1%</td>
</tr>
<tr>
<td>Crushing strength</td>
<td>82 N/mm</td>
<td>82 N/mm</td>
</tr>
<tr>
<td>BET Surface Area</td>
<td>25 m$^2$/g</td>
<td>22 m$^2$/g</td>
</tr>
</tbody>
</table>
2. KEY PROPERTIES OF $\beta$-SiC:

2.5. FLEXIBILITY OF THE PRODUCTION PROCESS

Si + C + Carbon binder

Extrusion

Spray Drying

Template coating

Shaped green bodies

Extrudates
Rings
Trilobes...

Micro Spheres

Open cell foams
3D Structures

Thermal Treatment
1. Company profile

2. Key properties of β-SiC

3. Current standard products
   3.1. General
   3.2. Typical values
   3.3. Purity

4. Capabilities for customization

5. Newly developed mesoC+™ Carbon support
3. CURRENT STANDARD PRODUCTS:
3.1. GENERAL

5 standard products available under extrudates form

---

**SiC1:**
- Commercial product

  *Stabilized product*
  - Can be supplied at >10 Mt/y
  - Available in 3 purity grades
  - Tight specifications for shape, pore volume, surface area, mechanical strength

**SiC2, SiC3, SiC4 and TiCSiC1:**
- Standard products

  *Can be supplied at tens of kg scale*
  - Available in 3 purity grades
  - Controlled properties
3. CURRENT STANDARD PRODUCTS:
3.2. TYPICAL VALUES

<table>
<thead>
<tr>
<th>SiC Grade</th>
<th>BET Surface Area</th>
<th>Microporous Surface Area</th>
<th>Pore Volume*</th>
<th>Crushing Strength**</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC1-E3-M</td>
<td>25 m²/g</td>
<td>&lt; 5 m²/g</td>
<td>0.40 cc/g</td>
<td>70 N/mm</td>
</tr>
<tr>
<td>SiC2-E3-M</td>
<td>25 m²/g</td>
<td>&lt; 5 m²/g</td>
<td>0.30 cc/g</td>
<td>40 N/mm</td>
</tr>
<tr>
<td>SiC3-E3-M</td>
<td>25 m²/g</td>
<td>&lt; 5 m²/g</td>
<td>0.55 cc/g</td>
<td>25 N/mm</td>
</tr>
<tr>
<td>SiC4-E3-M</td>
<td>30 m²/g</td>
<td>&lt; 5 m²/g</td>
<td>0.50 cc/g</td>
<td>50 N/mm</td>
</tr>
<tr>
<td>TiCSiC1-E3-M</td>
<td>90 m²/g</td>
<td>45 m²/g</td>
<td>0.35 cc/g</td>
<td>40 N/mm</td>
</tr>
</tbody>
</table>

* Measured by water absorption
** Grain method per ASTM D4179 & D6175

!!! NEW !!!
Highly mesoporous material
## 3. CURRENT STANDARD PRODUCTS:
### 3.3. PURITY

5 standard products available in 3 purity grades:

<table>
<thead>
<tr>
<th>Elemental analysis (ppm)</th>
<th>Purity grades</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>M</td>
</tr>
<tr>
<td>Fe</td>
<td>3 000</td>
</tr>
<tr>
<td>Al</td>
<td>1 000</td>
</tr>
<tr>
<td>Ca</td>
<td>400</td>
</tr>
<tr>
<td>Na</td>
<td>80</td>
</tr>
<tr>
<td>K</td>
<td>100</td>
</tr>
<tr>
<td>S</td>
<td>50</td>
</tr>
</tbody>
</table>

Typical values for SiC1, SiC2 and SiC4
1. Company profile

2. Key properties of β-SiC

3. Current standard products

4. Capabilities for customization
   4.1. Shapes
   4.2. Micro-structure
   4.3. Surface chemistry

5. Newly developed mesoC+™ Carbon support
4. CAPABILITIES FOR CUSTOMIZATION:
4.1. SHAPES

Current shapes easily available

*Fine Powders* \((d_{50}=3-5 \, \mu m)\)
*Extrudates*
*Open cell foams*

Demonstrated feasibility for

*Submicron powders* \((d_{50}< 0.5 \, \mu m)\)
*Grains* (50-800 \, \mu m)
*Micro-spheres* (100 \, \mu m range)
*Macro-spheres* (mm range)
*Tableting / pressed pieces*
4. CAPABILITIES FOR CUSTOMIZATION:

4.2. MICROSTRUCTURE

Customized surface area:
- 10 to 50 m²/g for pure SiC with no contribution of micropores
- Up to 130 m²/g for doped SiC with additional micropores

Customized porosity:
- Monomodal, bimodal or trimodal distribution
- Pore diameter from 10 nm to 10 µm
- Pore volume up to 1 cc/g
Thickness increase of amorphous SiO$_2$ layer:
- Via further oxidation of raw SiC
- Useful for zeolites synthesis with strong anchorage onto the carrier

Acid treatment:
- Removes surface impurities
- Changes in SiC surface chemistry can impact final catalytic performances

Mixed compounds to combine SiC and other component benefits:
- Composites: TiC-SiC, TiO$_2$-SiC, ZrO$_2$-SiC…
- Coatings: Al$_2$O$_3$, TiO$_2$, ZrO$_2$, SiO$_2$, Zeolite, C…
- Doping
1. Company profile

2. Key properties of β-SiC

3. Current standard products

4. Capabilities for customization

5. Newly developed mesoC+™ Carbon support
mesoC+™ is our new self-bonded synthetic carbon

**Key features and benefits**

**vs activated carbons:**
- improved mechanical strength
- well controlled and tunable shapes
- large volume of meso- and macropores

**vs SICAT SiC products:**
- higher BET surface area
- conventional surface chemistry
- cheaper at equivalent purity level

### Key Features and Benefits

<table>
<thead>
<tr>
<th>Test</th>
<th>mesoC+™</th>
<th>Competitor C pellets*</th>
<th>SICAT-SiC**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crushing strength (ASTM D4179)</td>
<td>40 N/mm</td>
<td>&lt; 10 N/mm</td>
<td>70 N/mm</td>
</tr>
<tr>
<td>Attrition (ASTM D4058)</td>
<td>0.4 %</td>
<td>&gt; 1.5 %</td>
<td>0.4 %</td>
</tr>
<tr>
<td>Tapped bed density</td>
<td>580 g/l</td>
<td>350-450 g/l</td>
<td>820 g/l</td>
</tr>
<tr>
<td>BET surface area (N2 sorption)</td>
<td>275 m²/g</td>
<td>800-1000 m²/g</td>
<td>30 m²/g</td>
</tr>
<tr>
<td>Pore volume 6-100 nm (Hg intrusion)</td>
<td>0.48 cc/g</td>
<td>&lt; 0.2 cc/g</td>
<td>0.29 cc/g</td>
</tr>
<tr>
<td>Total pore volume (Hg intrusion)</td>
<td>0.52 cc/g</td>
<td>0.40-0.70 cc/g</td>
<td>0.48 cc/g</td>
</tr>
</tbody>
</table>

*: average of values measured on 3 commercial pellets of activated carbon derived from coconut shell

**: for SiC1-E3-HP pellets

Technical presentation, August 2017
**mesoC+™: a combination of micro, meso and macropores**

- Micropores and mesopores generate high surface areas

  BET surface area = 275 m²/g  
  - 185 m²/g of micropore surface area  
  - 90 m²/g of external surface area

- Mesopores and macropores favor mass transfer and maximize the catalytic surface available for the reaction

**Typical values from N₂ isotherm**

**Typical values from Hg intrusion**
Current scale (pilot): 30 kg / batch

Shapes available:
- Pellets: 1 to 5 mm
- Trilobes: 0.8 and 1.6 mm
- Rings: $\varnothing_{\text{out}} = 5, 6, 8, 13$ mm
  $\varnothing_{\text{in}} = 3, 4, 5, 6, 7, 8$ mm
- Others on demand

Possible customization:
- Change of meso and macropore size distribution
- Increase of BET through activation
**SICAT (Administration)**
11 rue de le Ville l’Evêque
75008 Paris, France

**SICAT (R&D)**
20 Place des Halles
67000 Strasbourg, France

**ACM GmbH (Production)**
Industriestraße 1, B310
77731 Willstätt, Germany

*Tel: +49 7852 8 1150*
*Email: info@sicatcatalyst.com*
*Website: www.sicatcatalyst.com*