Synthesis of Silicon Carbide

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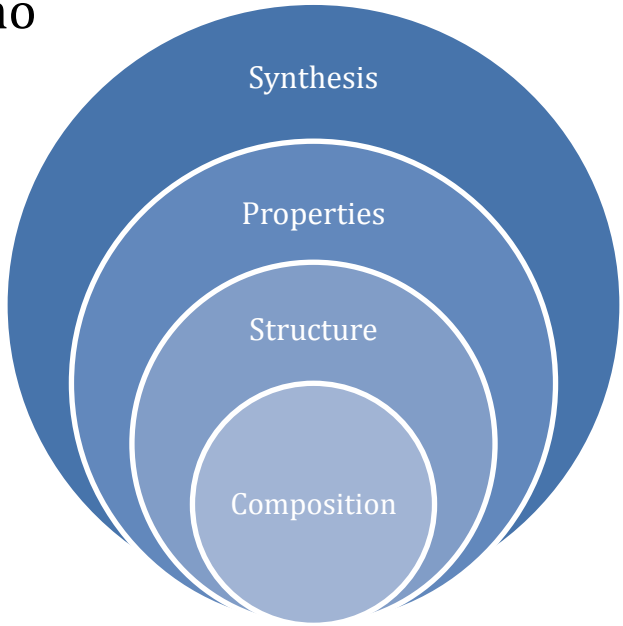
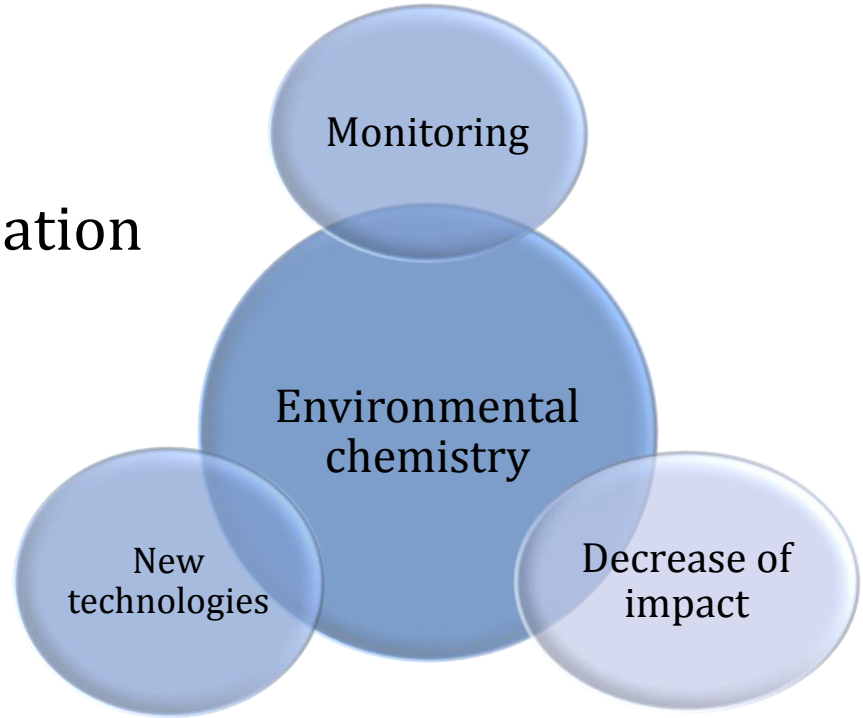
Research topics in MATCHEM

- Synthesis of advanced materials
  - Layers, bulk; inorganic, organic; micro, nano
- Synthesis and chemistry of polymers
  - Commercial, novel
- Electrochemistry and corrosion
  - New measuring methods; active, passive
- Inorganic biochemistry
  - Biochemistry of metal complexes



Research topics in ENVCHEM

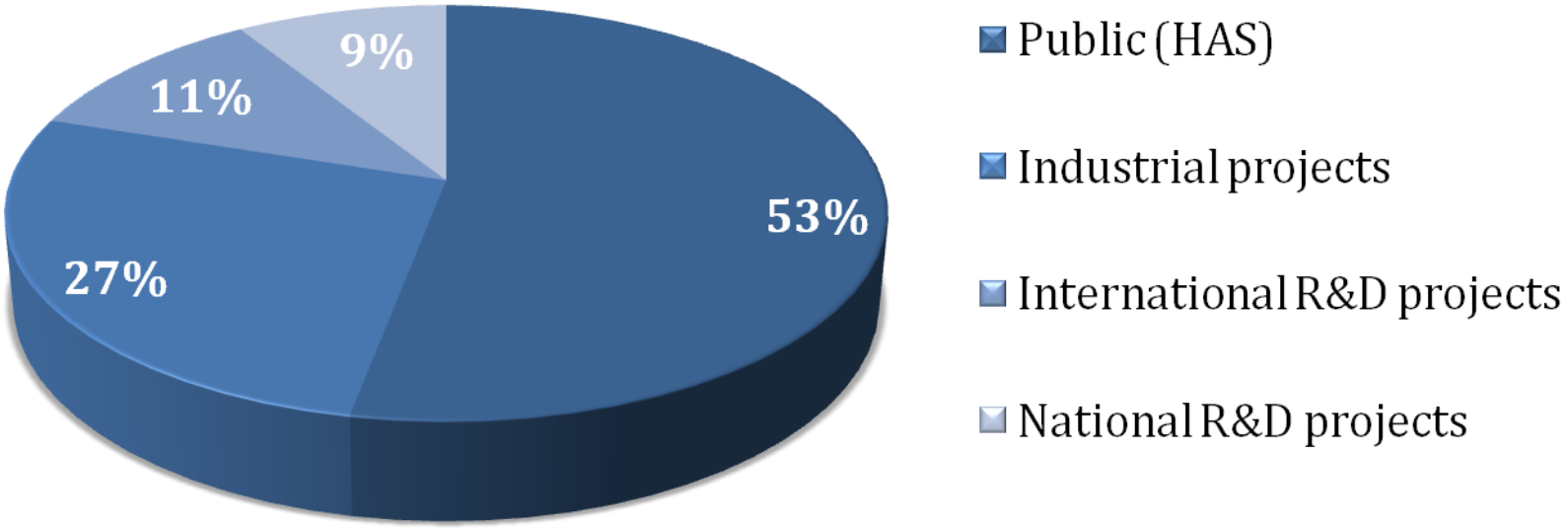
- Environmental monitoring and analysis
- Renewable energy
  - Biomass and solar
- Waste processing and remediation
  - Hazardous organic & inorganic
  - Chemical & thermal
  - Soil, water, air
- New technologies
  - Low environmental impact



Some figures (2010)

- Staff number: 85
- Researchers: 52 (<35 years: 26)
- Annual budget: 2,500,000 EUR

Funding (2010)



Properties, production, application

SILICON CARBIDE

Properties of silicon carbide

- High strength and hardness (21-25 GPa) at high T
- Low thermal expansion ($5 \cdot 10^{-6} K^{-1}$)
- Good thermal conductivity ($50\ W \cdot m^{-1} \cdot K^{-1}$)
- Thermal shock resistance
- Thermal and chemical stability
- Resistant to
  - Oxidation even at high T
  - Acids and alkalis, molten metals
- Semi-conductive properties

Applications of silicon carbide

- Self-sharpening abrasives, cutting tools,
- Refractory materials
- Diesel particulate filters
- Membranes for water purification
- Filters for hot gas cleaning
- Heat exchangers, heating elements
- Dielectric coatings
- Blue LEDs
- Varistors

Production of silicon carbide

- **Microsized SiC**
  - Acheson process: CR of SiO_2 at 2200-2400 ^oC ⇨ α-SiC
  - Rapid carbothermal synthesis ⇨ α-SiC
  - High energy mechanical milling ⇨ β-SiC
- **Micro- and nanosized SiC**
  - Pyrolysis of $SiCH_3Cl_3$ or TPS at 1000-1500 ^oC ⇨ α- and β-SiC
  - Sol-gel synthesis from Si(OR)4 ⇨ α- and β-SiC
  - Vapor phase reactions of SiH_4 or $SiCl_4$ and CH_4 or C_2H_4 ⇨ β-SiC

Novel synthesis of silicon carbide

TYGRE PROJECT

SiC from waste tyre gasification residue

ENEA

- Horizontal furnace (HF)
- >1400°C
- t > 30 min

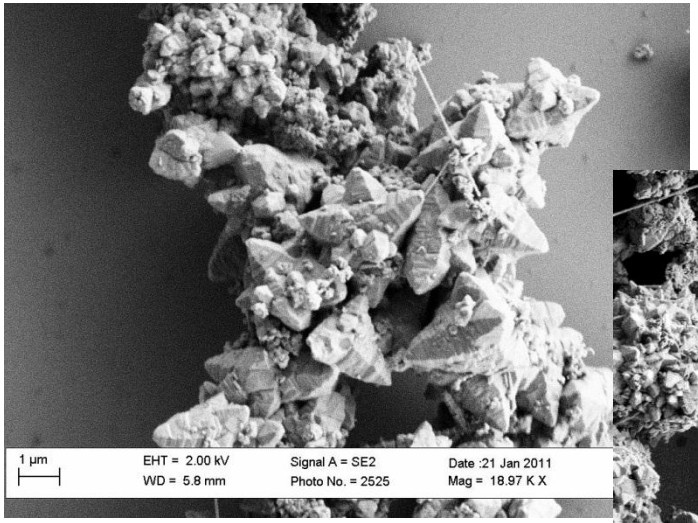
IMEC

- Thermal plasma reactor (TPR)
- > 2000 °C
- Several seconds

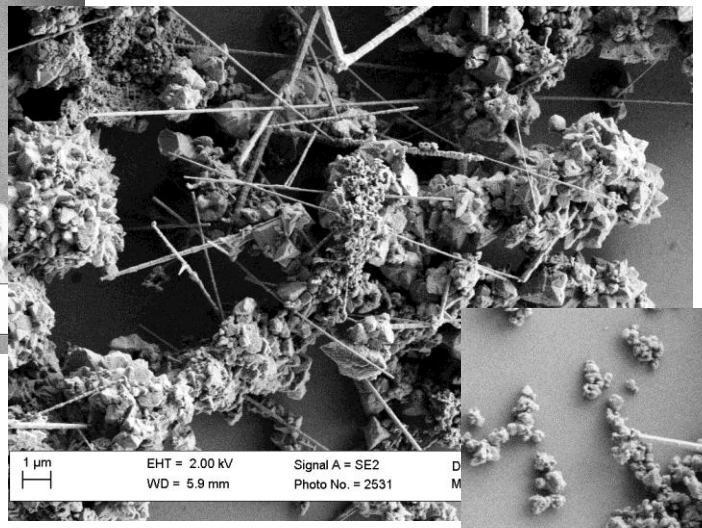
Conditions of synthesis in HF

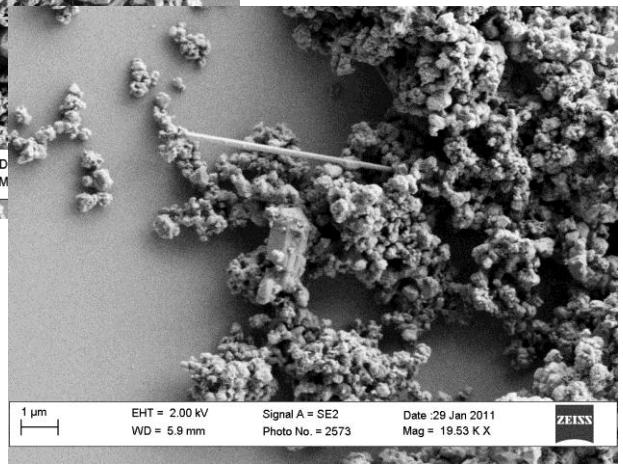
- **Carbon sources**
  - Chars from pyrolysis/gasification
  - Active carbon
- **Silica sources**
  - Alternative Silica Sources
- **Process parameters**
  - Temperature
  - Residence time
  - Composition of the gas phase

Morphology of SiC



From as-produced char



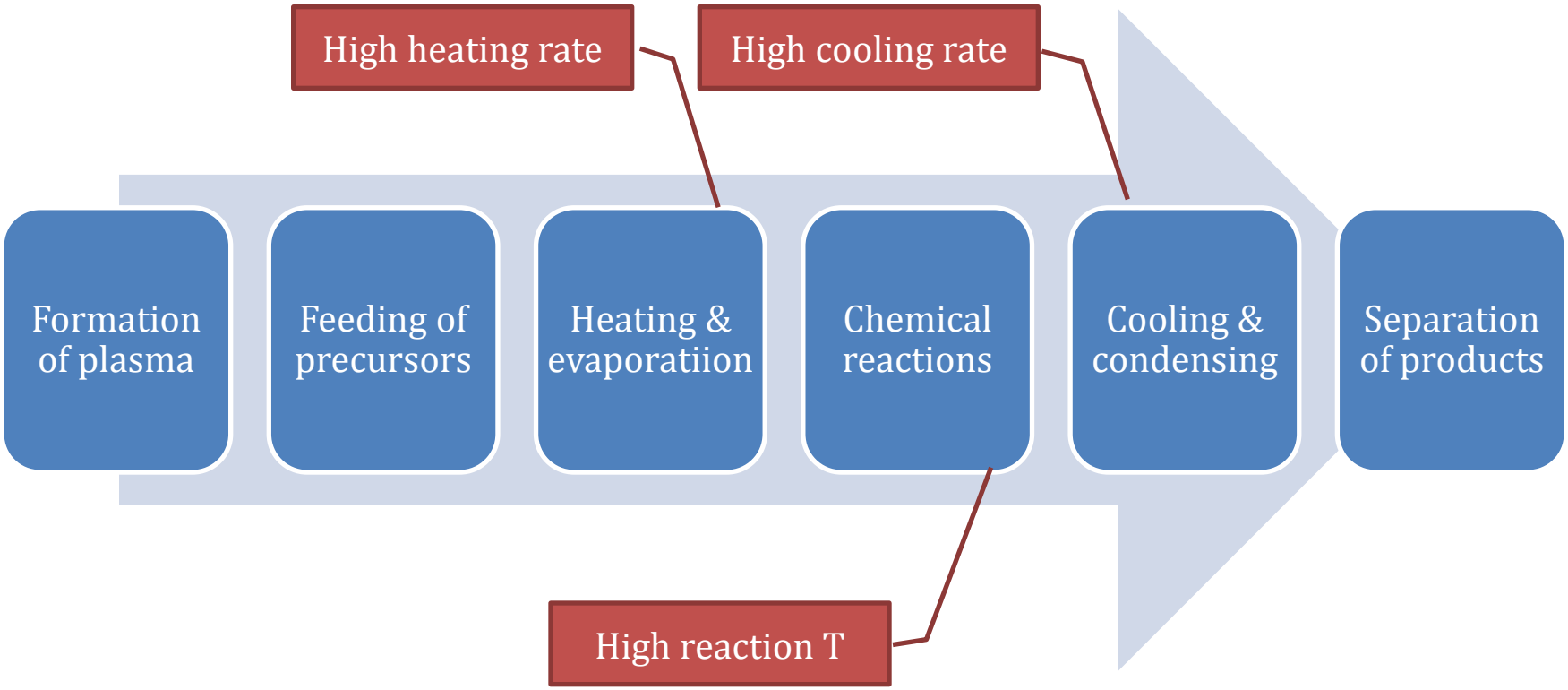


From purified char

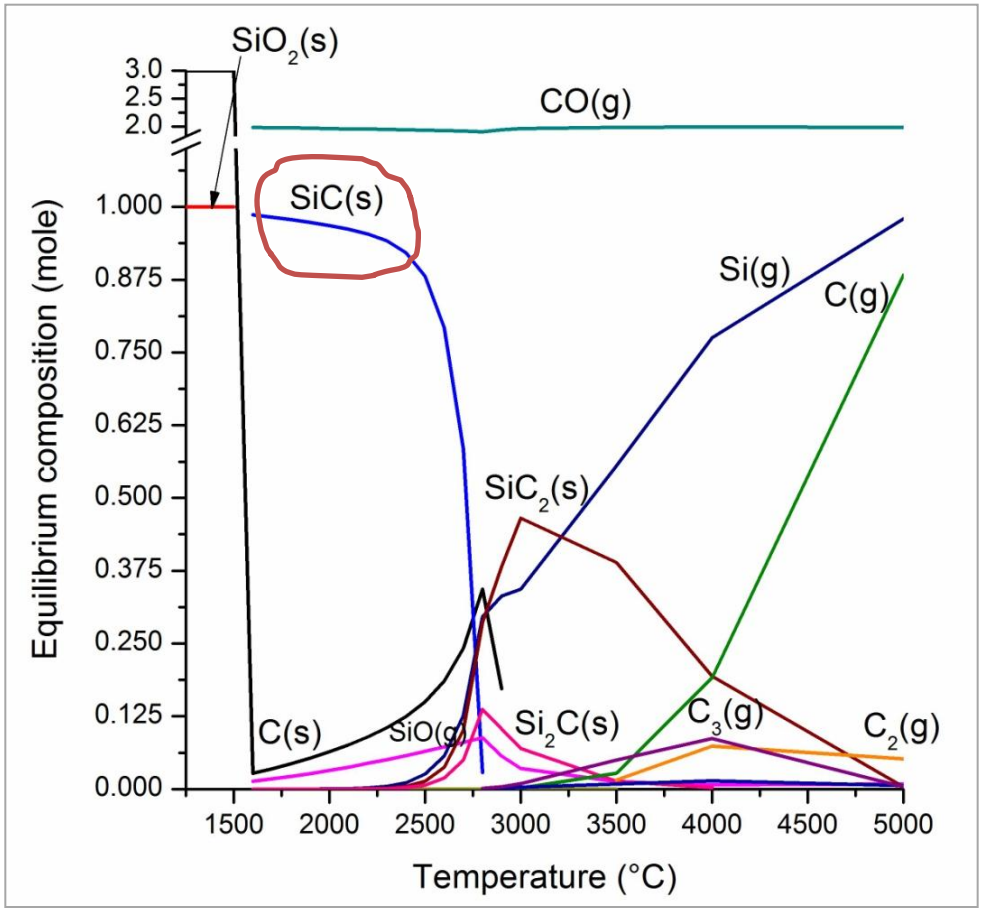
Summary of tests in HF

- **Synthesis conditions**
  - Carbon : silica molar ratio > 3:1
  - T >1400°C, t > 30 min
- **SiC yield**
  - 90 – 95 % of theoretical yield
- **Properties of products**
  - Microcrystalline, mainly β-SiC particles and fibers in Ar
  - Si_3N_4/SiC composite in N_2

Particle synthesis in TPR



TD calculations for TPR



Formation of SiC is favored from 1600 to 2500 °C

Conditions of synthesis in TPR

- **Carbon sources**
  - Char from tyre pyrolysis/gasification
  - Active carbon
  - Commercial graphite
- **Silica sources**
  - Alternative Silica Sources
  - Fly ash
- **Parameters studied**
  - C : SiO_2 molar ratio
  - Feed rate of precursors
  - Plasma gas composition
  - Cooling rate of products

Chemical composition of SiC from TPR

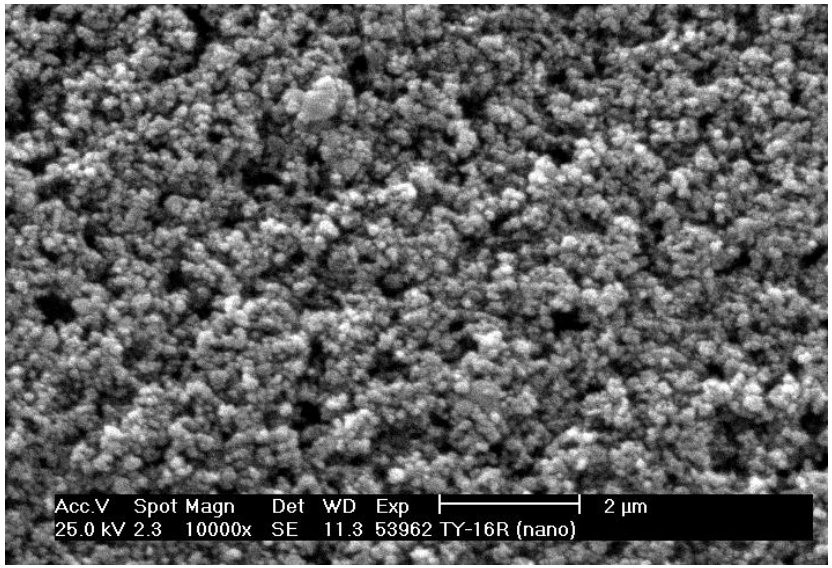
Bulk (wt%)

- Oxygen: 4.0
- Carbon: 32.8
- Al, Ca, Mg, Na, Si, Zn: about 5

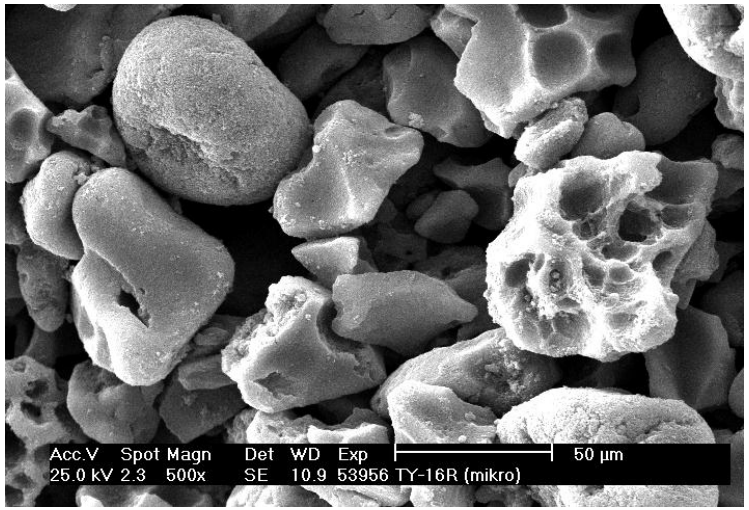
Surface (wt%)

- O: 12.5
- C: 35.1
- Si: 47.1

Morphology of SiC from TPR



Nanosized (main fraction)



Microsized (minor fraction)

Summary of tests in TPR

- **Synthesis conditions**
  - Carbon : silica molar ratio: > 4:1
  - Plasma gas: Ar + He
  - Specific energy: 4-5 kWh/kg
- **SiO_2 conversion**
  - Above 70 %
- **Properties of products**
  - Nanocrystalline, mainly β-SiC particles
  - Amorphous Si_3N_4 in the presence of NH_3

Comparison of HF and TPR

Parameter	HF	TPR
Yield (relative, wt%)	> 90%	> 70%
By-products	Low SiO_2, C	SiO_2, C, Si, Zn
Phases (purified)	β-SiC	(α+β)-SiC, Si, C
Morphology	Microcrystals and fibers	Mainly nanocrystals
Process	Simple, conventional	Special, innovative
Sintering	Conventional	Development needed
Application	Membrane	Membrane, others
Price (Aldrich)	100 EUR/kg	4000 EUR/kg

Conclusions

- Waste tyre pyrolysis residue ⇨ high grade silicon carbide
- Both HF and TPR are proper tools
- Properties depend on
  - Quality of raw materials
  - Synthesis conditions
- In TPR nanosized SiC
- High added value
- Scale-up in progress