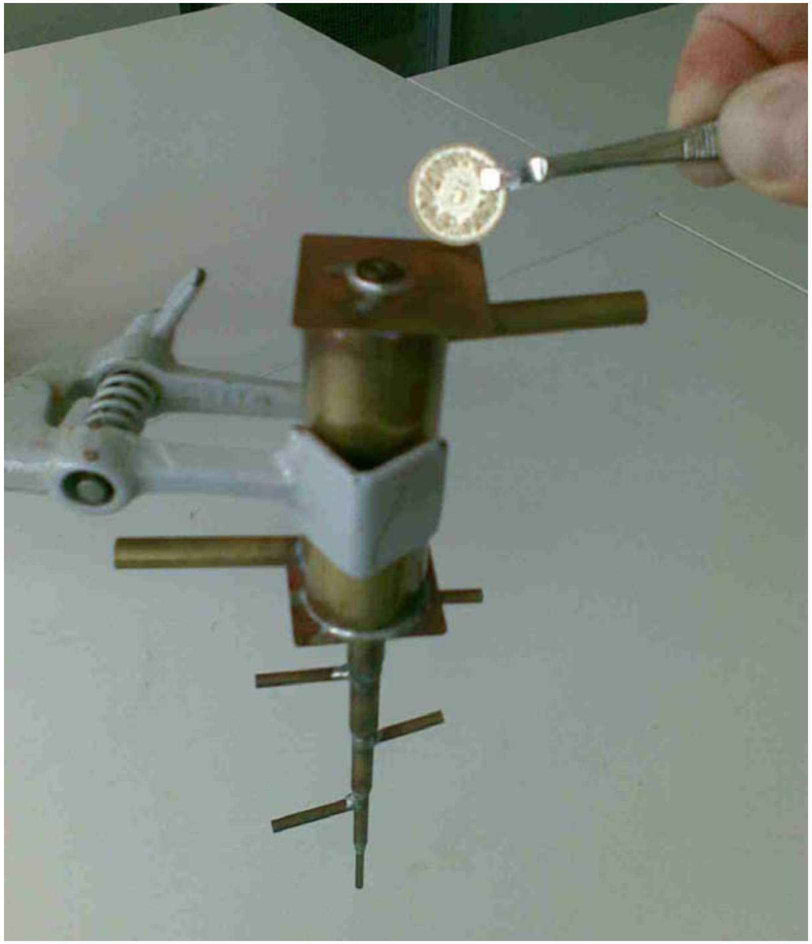
Flame Aerosol Synthesis

From Lab-Scale Experiments to Pilot Plant Production

Karsten Wegner

Wegner Consulting & ETH Zürich

How everything started...



Prototype diffusion flame microreactor

Material: copper, welded

Fabrication: hand-made in Cincinnati (OH)

Characteristics:
6 “concentric” tubes 1/10”, 1/6”, 7/32”, ...

The Swiss response:

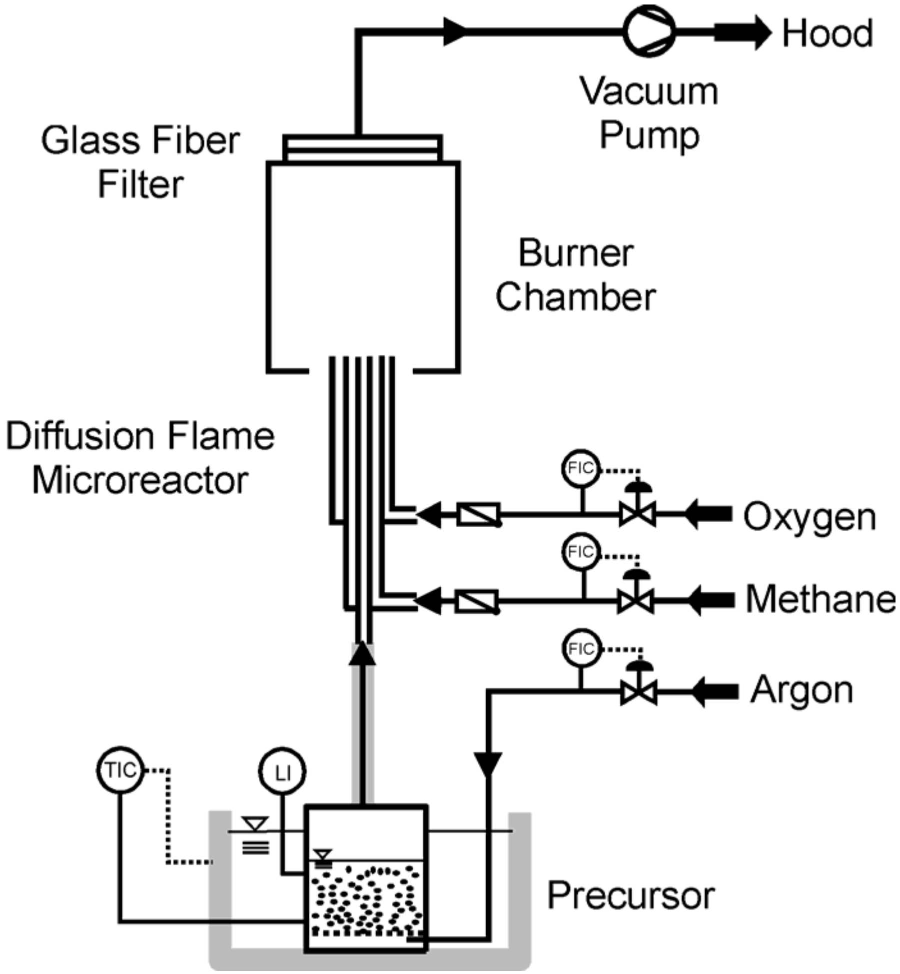
Diffusion flame microreactor

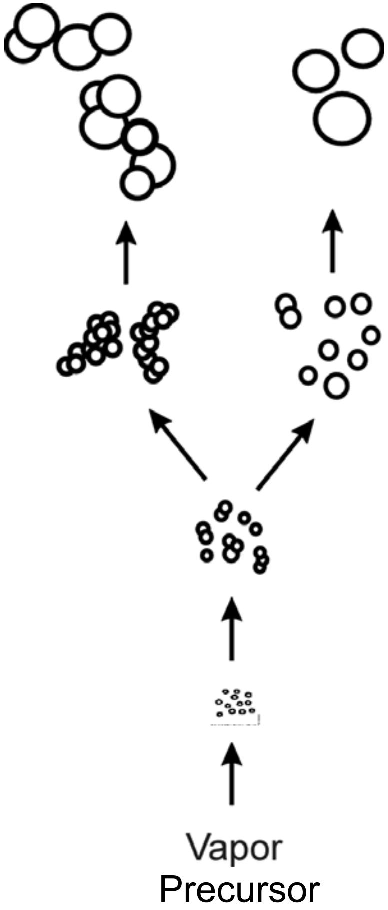
Material: stainless steel 1.4435

Fabrication: machined at ETH workshop

Characteristics:
6 concentric! tubes: 2.54mm, 4.23mm, 5.56mm,...

Flame aerosol synthesis set-up





Product

Collection

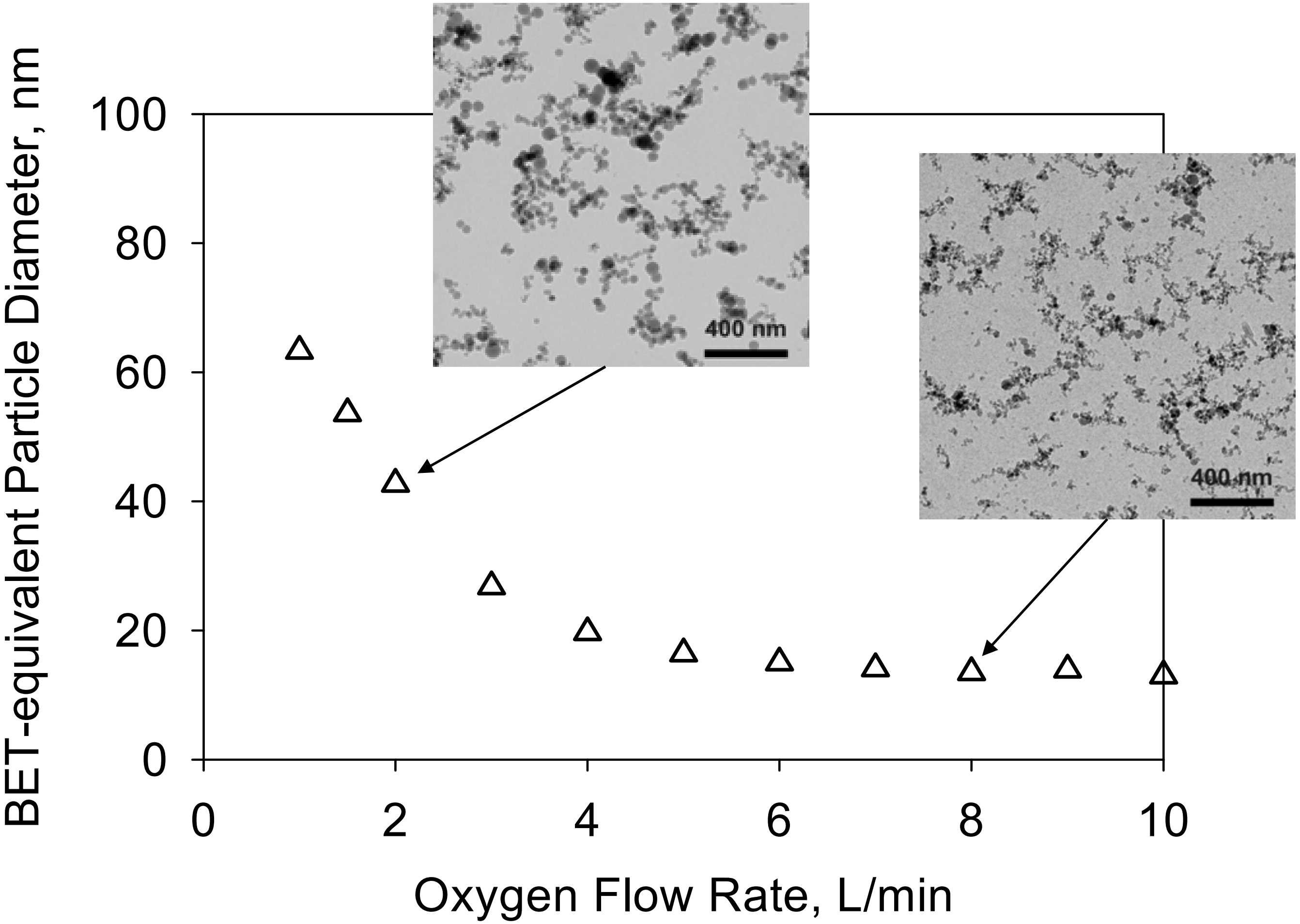
Collision

Sintering

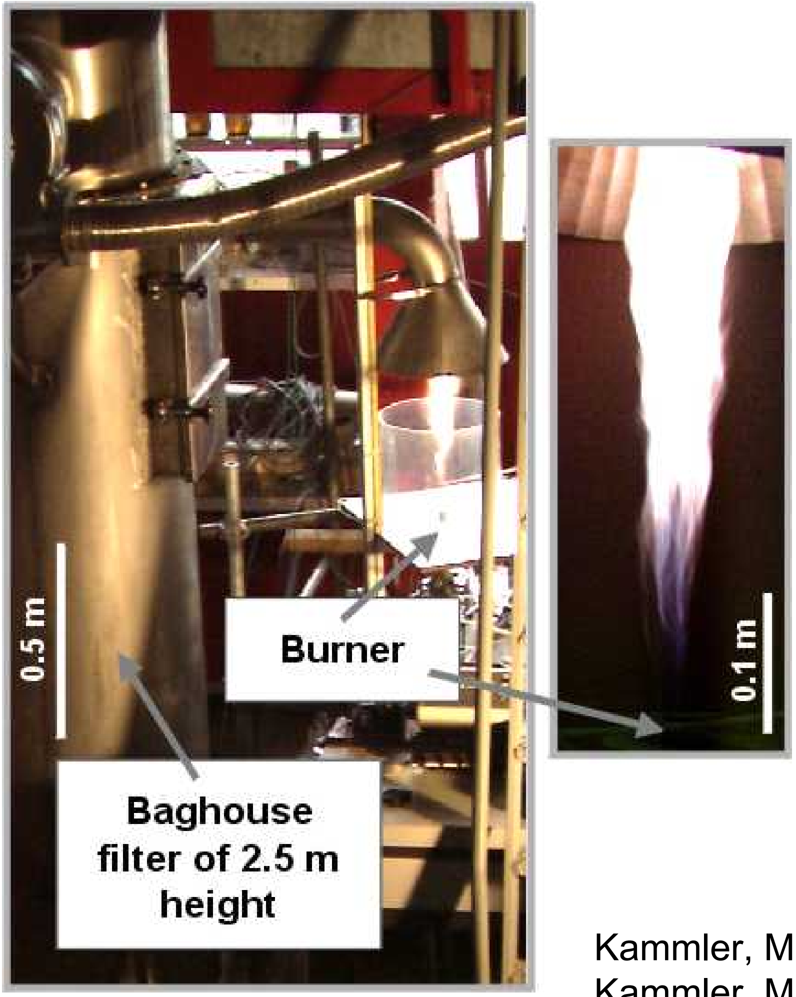
Reaction

The first nanoparticles at ETH-PTL

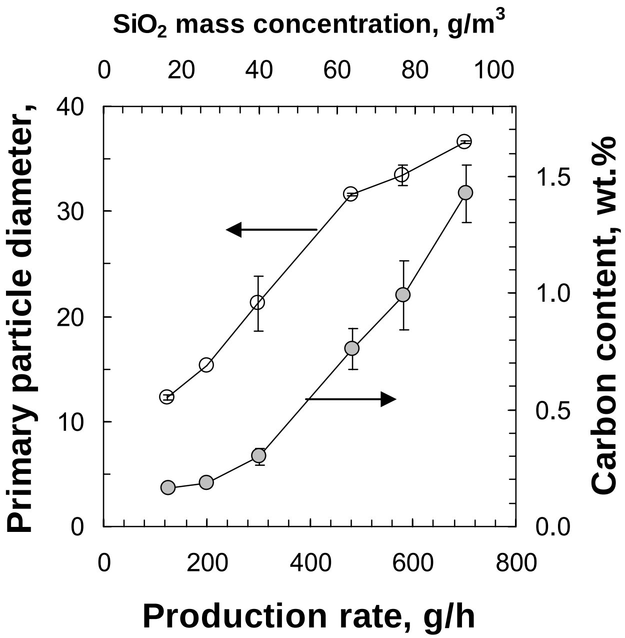
~5 g/h silica from hexamethyldisiloxane (HMDSO)



Early pilot-scale production

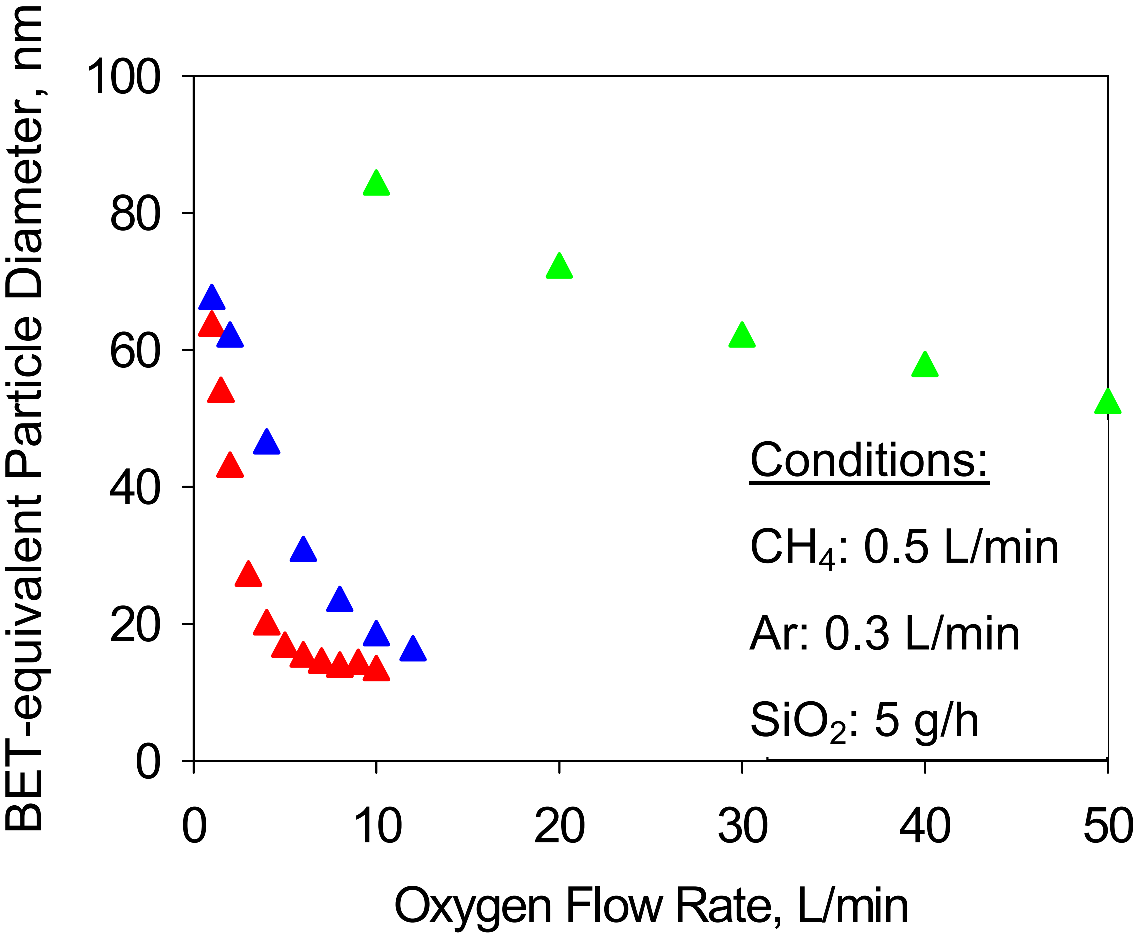


Hydrogen-oxygen diffusion burner obtained from German Aerospace Research Center (DLR)



Kammler, Mueller, Senn, Pratsinis, *AIChE J.* **47**, 1533 (2001)
Kammler, Mädler, Pratsinis, *Chem. Eng. Technol.* 24, 583 (2001)

Burner Operation Lines

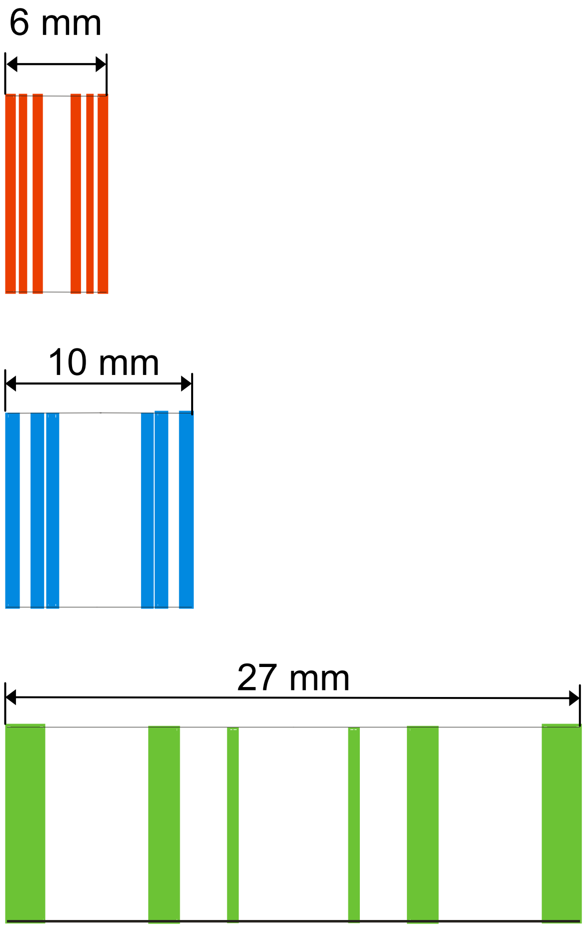


Conditions:

CH_4: 0.5 L/min

Ar: 0.3 L/min

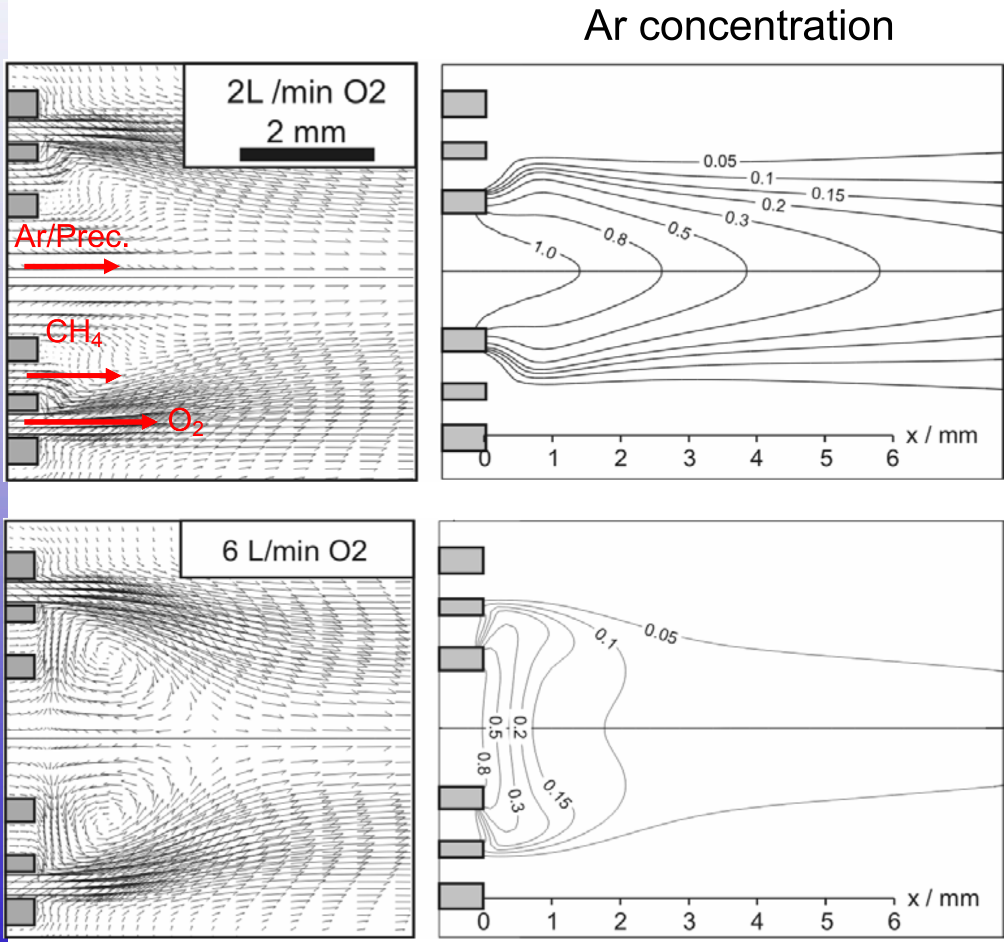
SiO_2: 5 g/h



Wegner and Pratsinis, *Chem. Eng. Sci.* **58**, 4581 (2003)

Coaxial Jet Mixing

Cold flow CFD profiles:



Diffusion Flames:

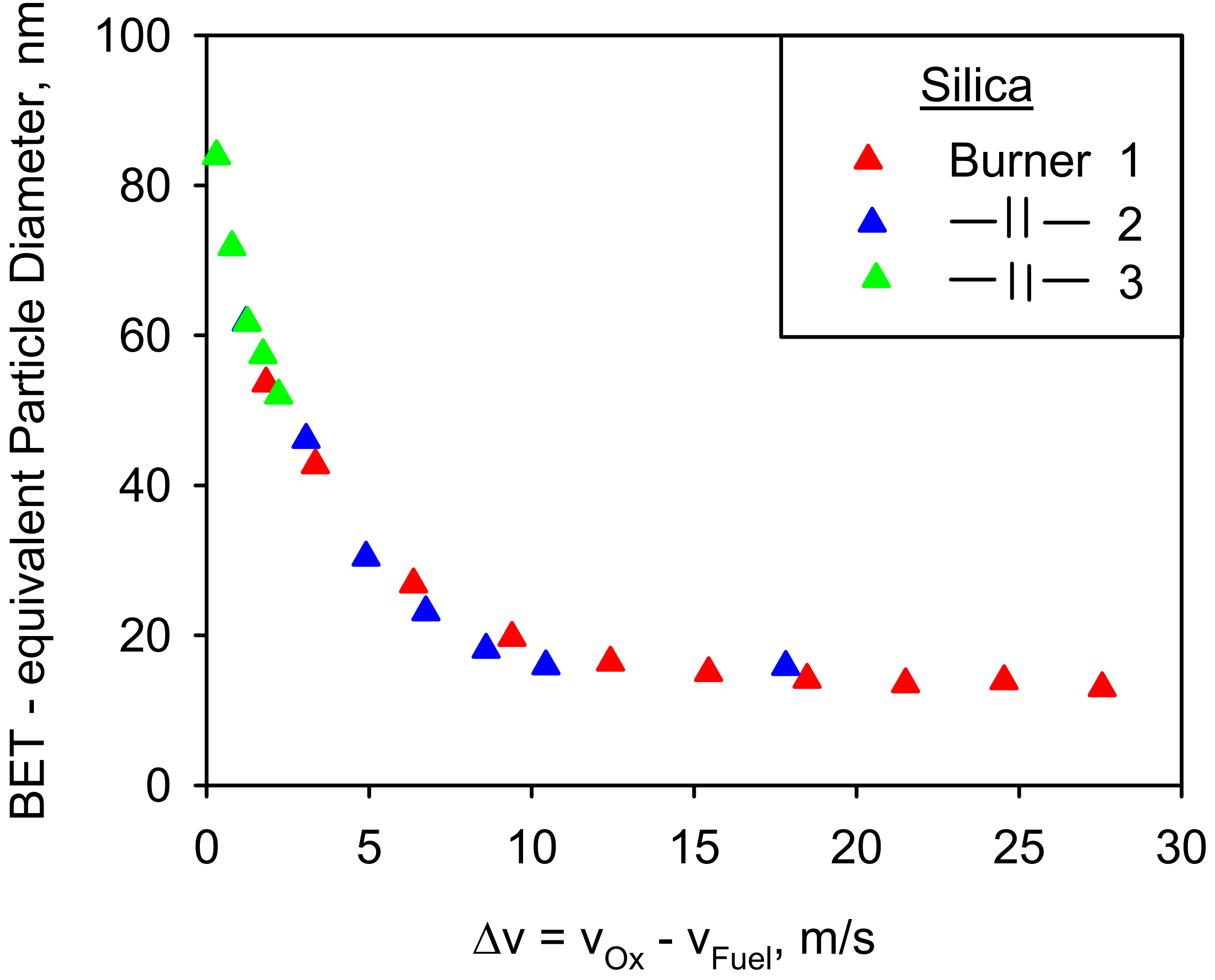
$$t_{reaction} \ll t_{mixing}$$

Particle Formation upon mixing of precursor and oxidant at shear layer.

Similar reactant mixing for similar velocity difference $\Delta v = v_{Ox} - v_{Fuel}$

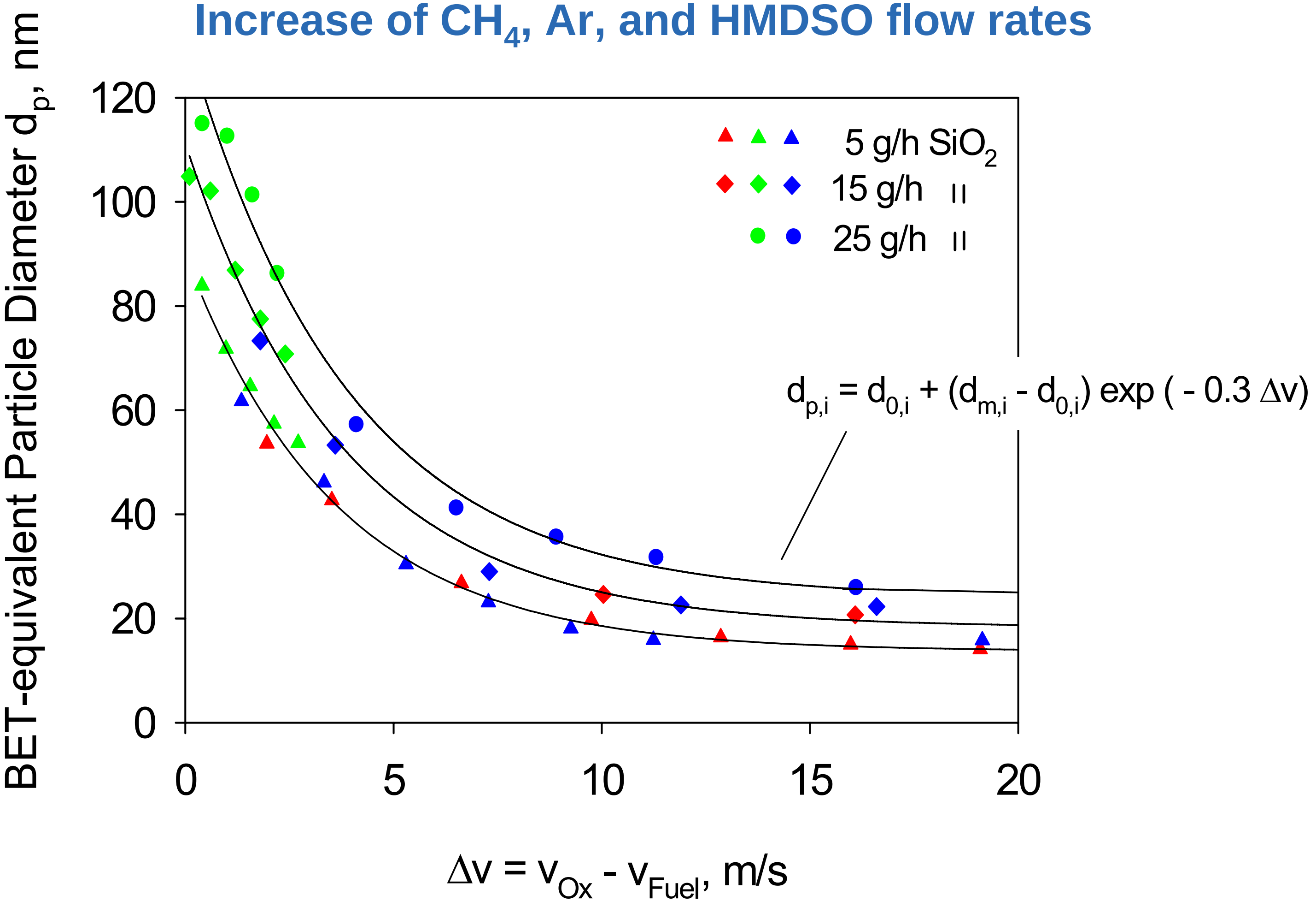
$$\underbrace{v_{Ar+Prec.} \approx v_{CH_4}}_{v_{Fuel}} \leq v_{Ox}$$

Single Operation Line $d_p = f(\Delta v)$



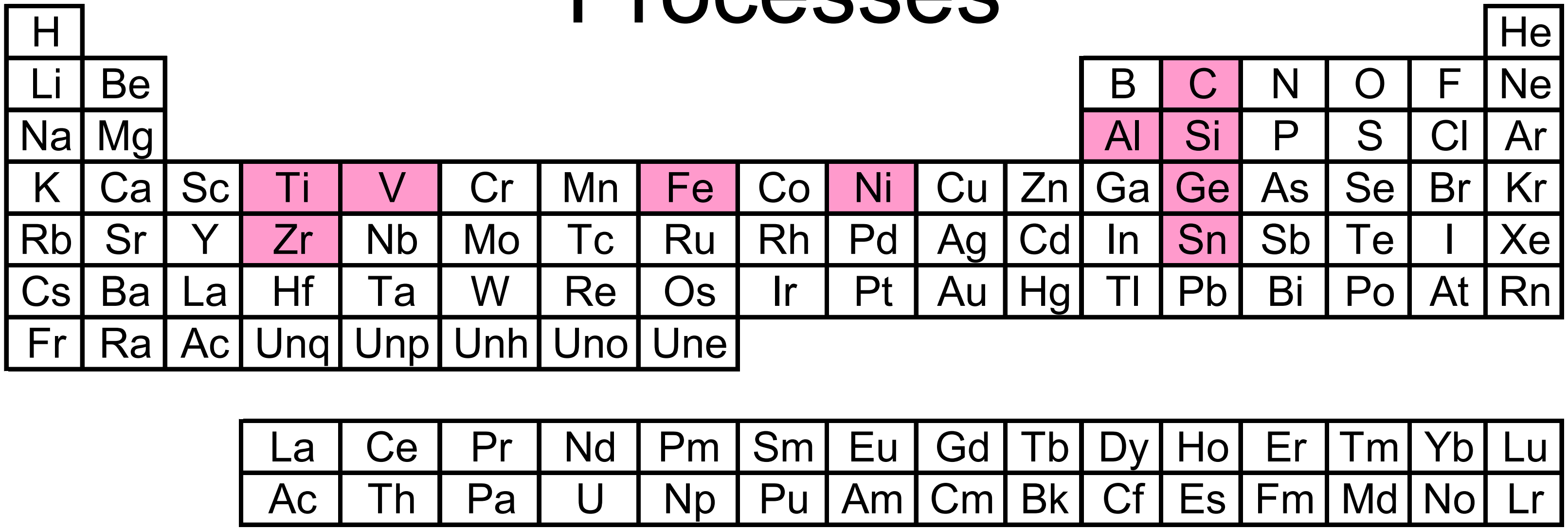
Scaling the SiO_2 Production Rate

Increase of CH_4, Ar, and HMDSO flow rates



Wegner and Pratsinis, *Chem. Eng. Sci.* **58**, 4581 (2003)

Products of Conventional Flame Processes



H																	He
Li	Be											B	**C**	N	O	F	Ne
Na	Mg											**Al**	**Si**	P	S	Cl	Ar
K	Ca	Sc	**Ti**	**V**	Cr	Mn	**Fe**	Co	**Ni**	Cu	Zn	Ga	**Ge**	As	Se	Br	Kr
Rb	Sr	Y	**Zr**	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	**Sn**	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Unq	Unp	Unh	Uno	Une										

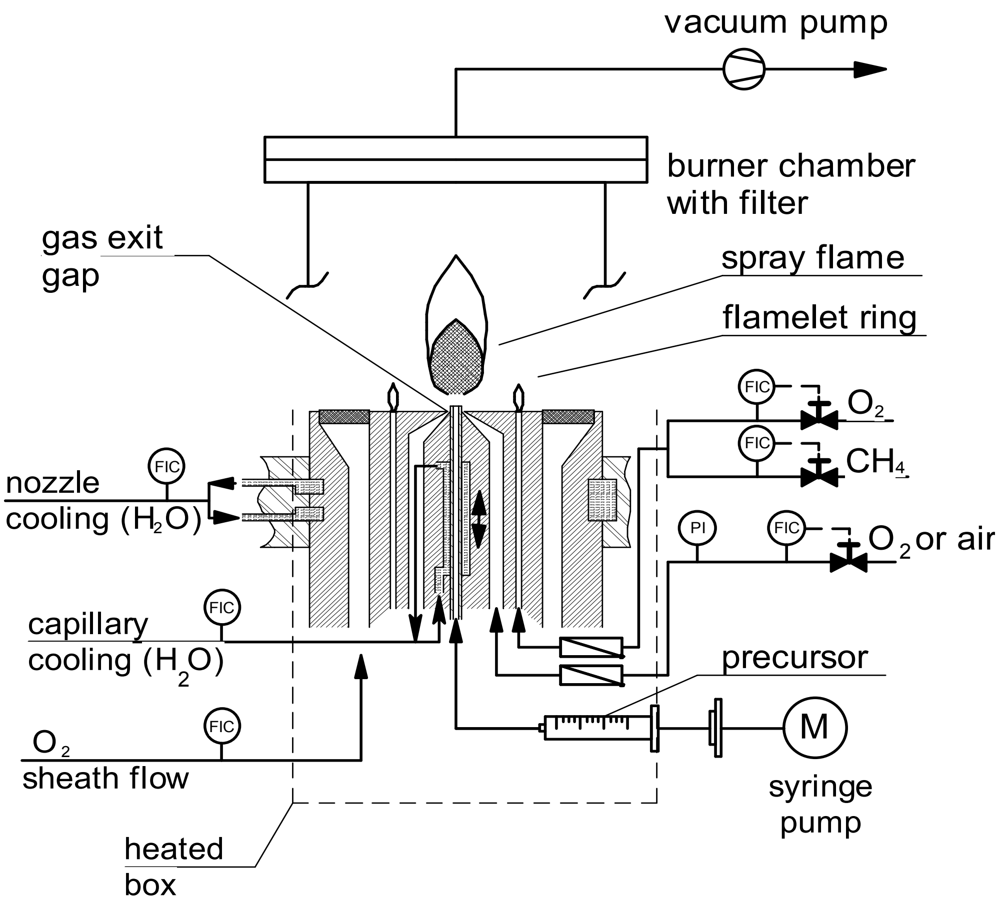
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

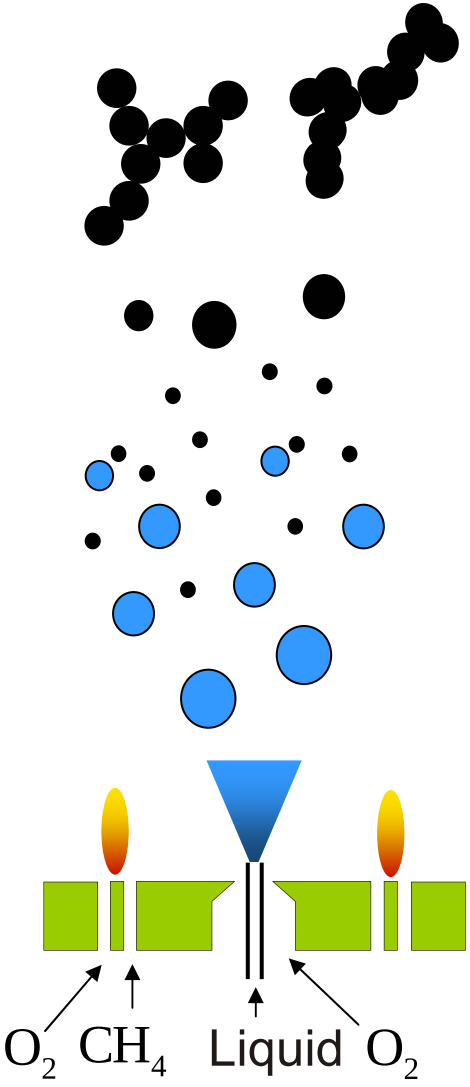
Few metal oxides accessible due to limited availability of low-cost precursors with high vapor pressure at moderate Temp.

At ETH-PTL: SiO_2, TiO_2 - but also first flame-made catalysts: vanadia/titania at small and pilot scale.

Stark, Wegner, Pratsinis, Baiker, *J. Catal.* **197**, 182 (2001)
Stark, Baiker, Pratsinis, *Part. Part. Sys. Char.* **19**, 306 (2002)

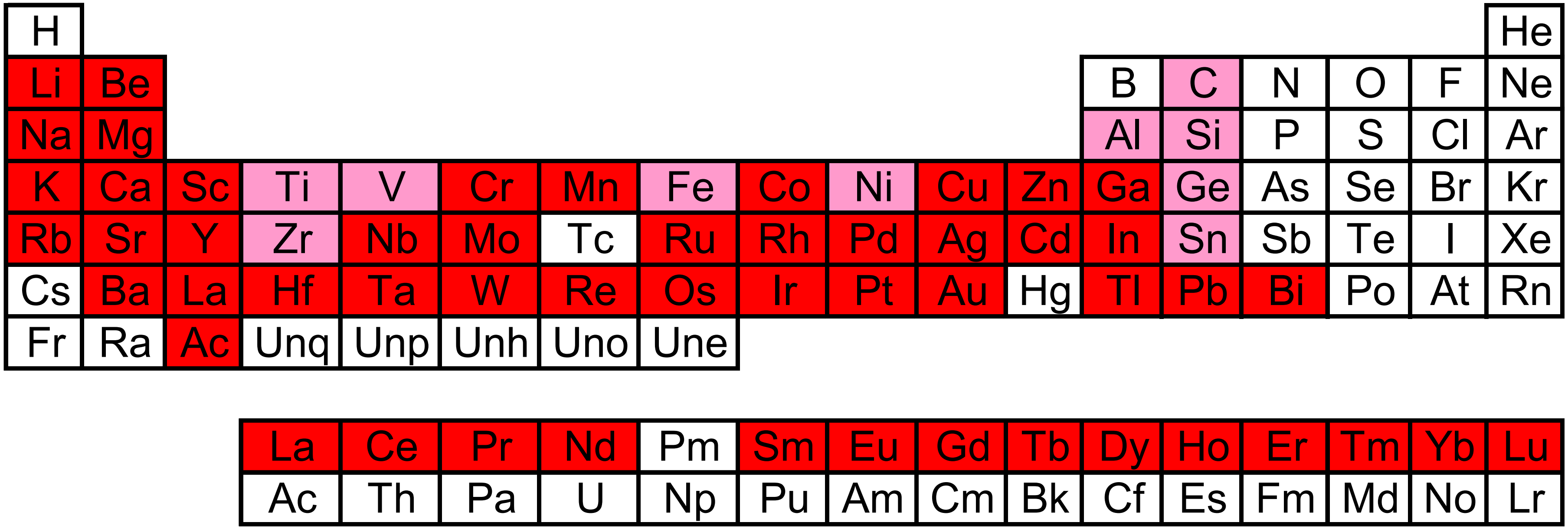
Flame Spray Pyrolysis (FSP) Technology





Mädler, Kammler, Mueller, Pratsinis, *J. Aerosol Sci.* **33**, 369 (2002).

Accessible Products by FSP



H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Unq	Unp	Unh	Uno	Une										

La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

🟥 Noble metals and oxides of almost all periodic table elements

▨ Flame synthesis using vaporous precursors

Benefits of the FSP Process

Broad range of product materials

Multi-component particles

Good control of particle properties

High purity powders

Thermally stable powders

Environmentally friendly process

Short process chain

Standard equipment and materials

Low cost energy source

Recent review article: Strobel and Pratsinis, *J. Mater. Chem.* **17**, 4743 (2007).

Scale-up from 10 to ~500 g/h

ETH Lab-Scale

ZÜNDENDE PROJEKTE FÜR MASSGE-SCHNEIDERTE NANOPARTIKEL.

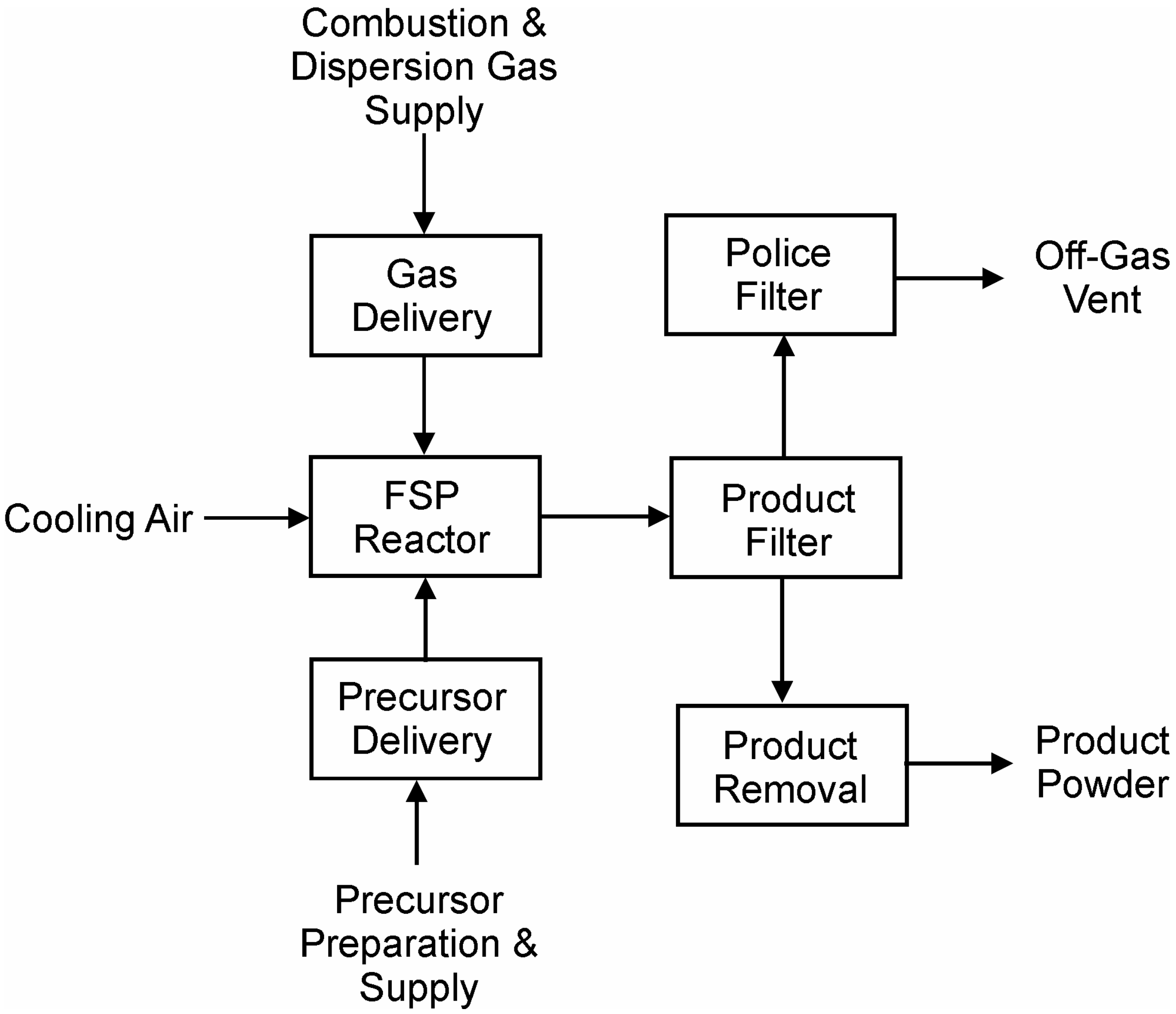
JAHRESBERICHT 2003

ETH Pilot System

Mueller, Mädler, Pratsinis, *Chem. Eng. Sci.* **58**, 1969 (2003).
Mueller, Jossen, Pratsinis, *J. Am. Ceram. Soc.* **87**, 197 (2004).

Tokyo Nanotech 2008

Process Flow Diagram


Combustion & Dispersion Gas Supply

Gas Delivery

Cooling Air

FSP Reactor

Precursor Delivery

Precursor Preparation & Supply

Product Filter

Police Filter

Off-Gas Vent

Product Removal

Product Powder

1 kg/h Pilot Plant at FlamePowders

5 kg ZnO nanoparticles

Process Design Considerations

Product specs., production rate, raw materials
Purity requirements
Available infrastructure

Reactant Delivery

Chemicals used?
Batch/continuous?
Precursor preparation?
Precursor stability?
Quality control?
Cleanability?
Safety!

FSP Reactor

Flow-/production rates?
Auxiliary gases?
Stability of combustion?
Process Control?
Energy utilisation
CO_2 reduction
Containment!
Safety!

Filter

Filtration area?
Flow field
Nanopowder discharge
Off-gas treatment
Product or clean side filter change?
Product change?
Containment! Safety!

Nanoparticle containment and safety concept.
Process automatization

100 g/h Pilot Plant for NanoCentral

Location: Johnson Matthey Research Center, UK

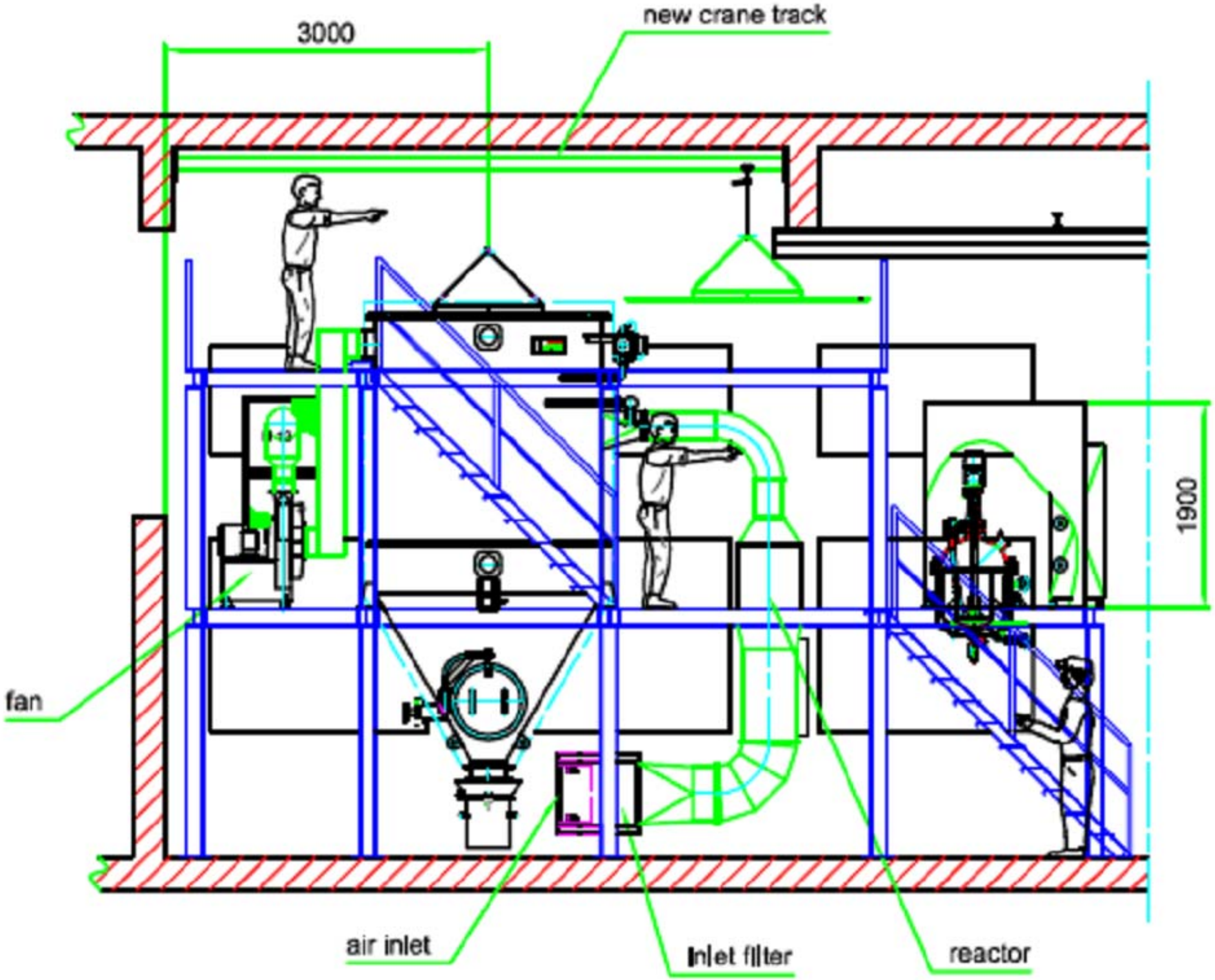
www.nanocentral.eu

Automated FSP Unit at L’Urederra

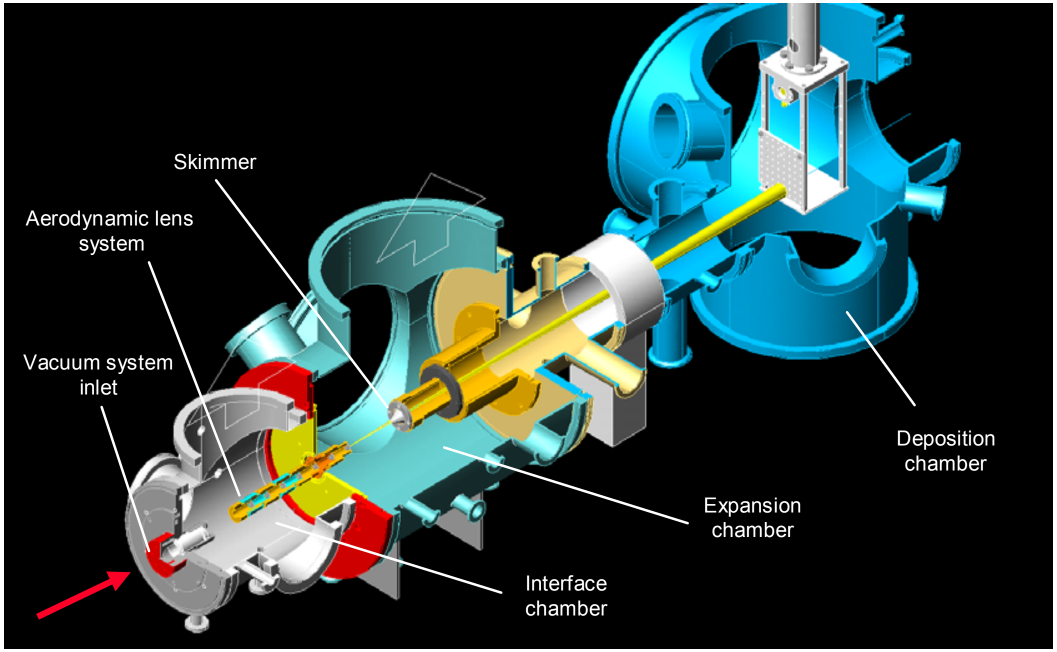
Fundación L’Urederra
Los Arcos (Navarra), Spain

Production rate 0.5 – 1.0 kg/h, continuous operation
Process control by PLC

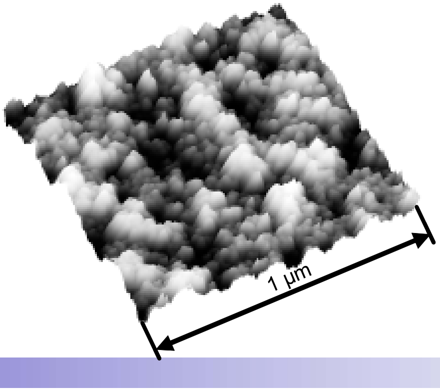
Under construction: 1 kg/h unit

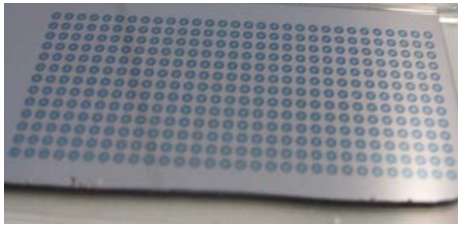


Back to the small scale:
Combined FSP-cluster beam deposition



Nanostructured particulate films





Patterned deposition

Thank you very much!

Questions?