Combustion Synthesis of Materials
2. How to closely control aerosol particle characteristics

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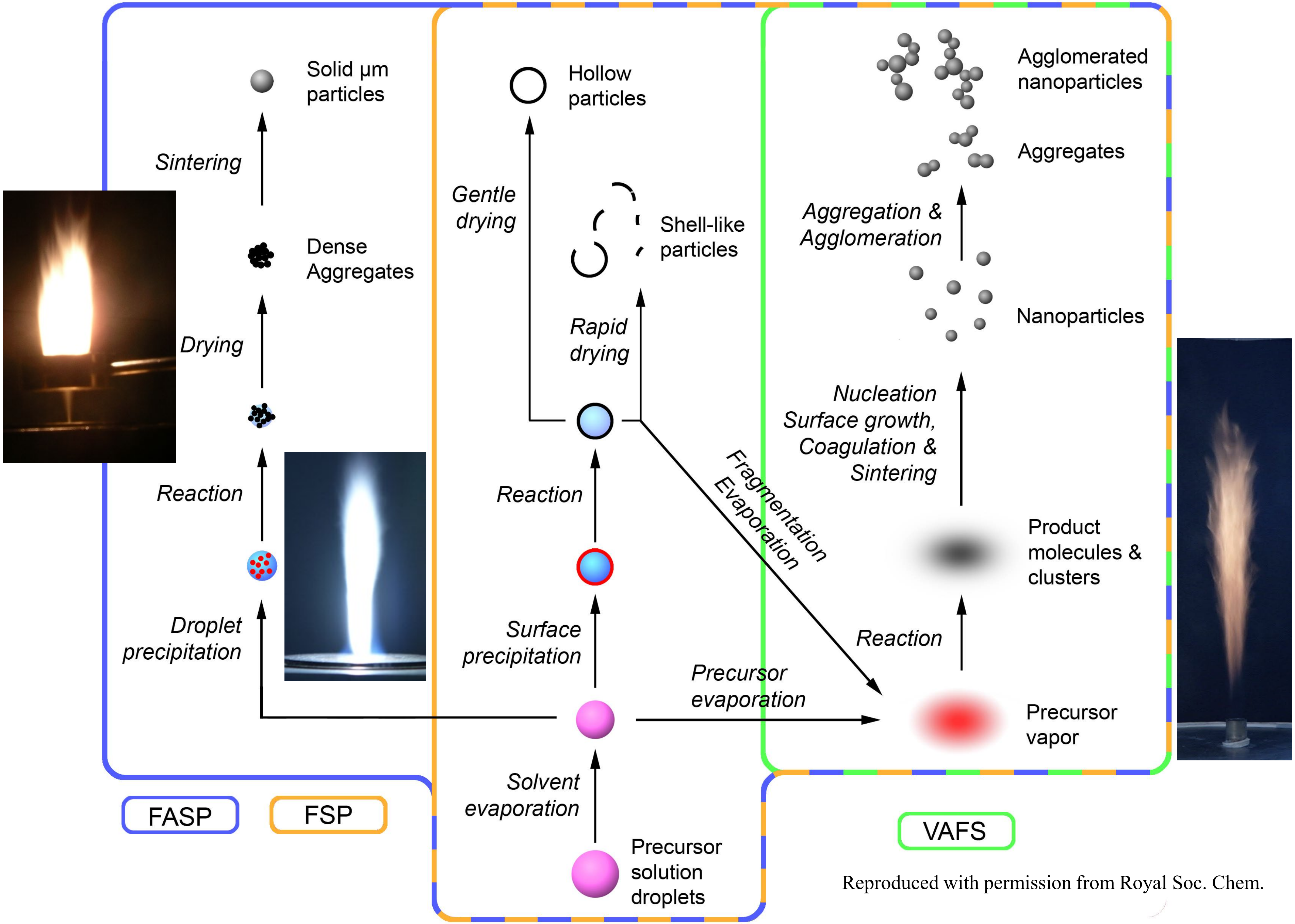
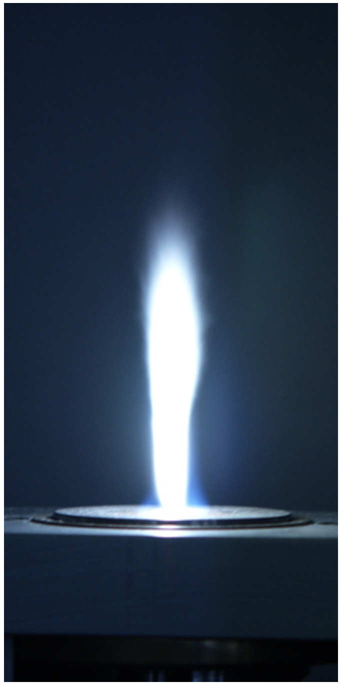


R. Strobel, SEP Flame aerosol synthesis of smart nanostructured materials. J. Mater. Chem., 17, 4743 - 4756 (2007).

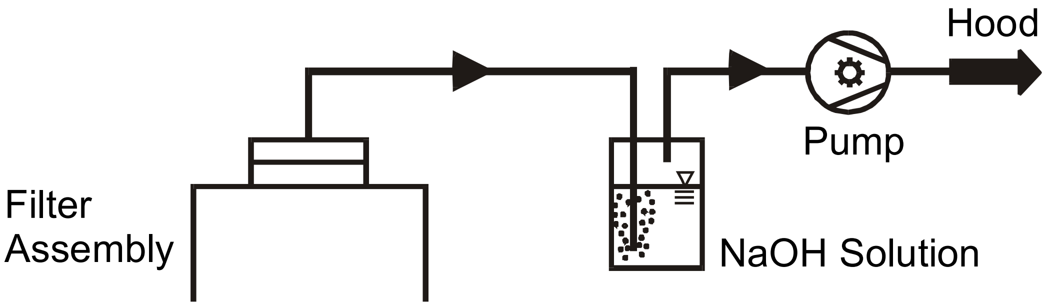
Catalyst Synthesis by Flame Spray Pyrolysis (FSP)

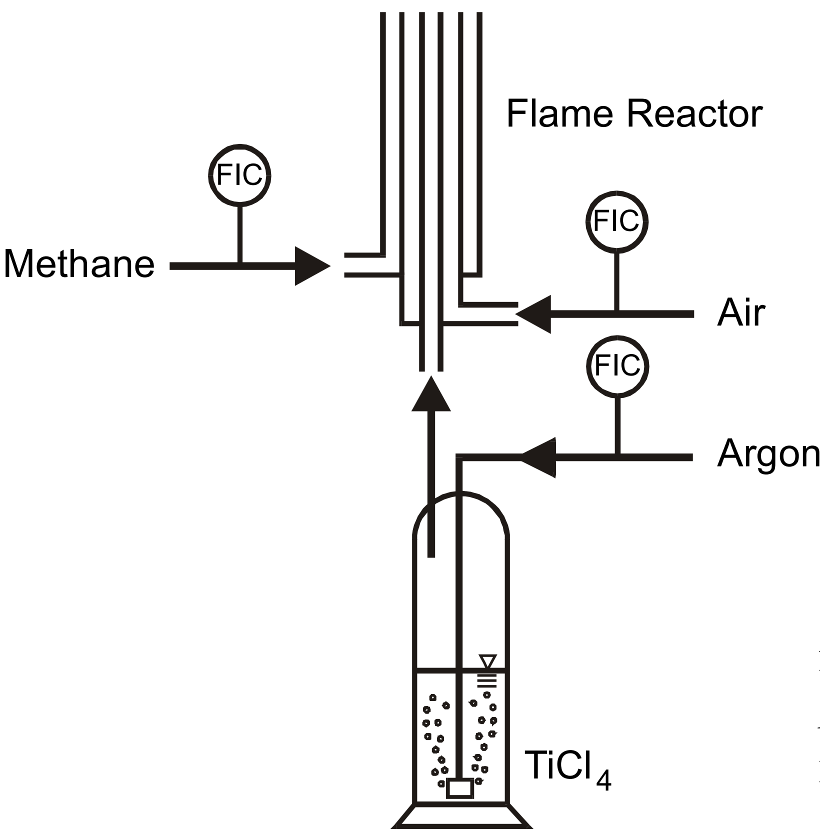
Strobel, Stark, Mädler, SEP, Baiker, *J. Catal.* **213**, 296-304 (2003)

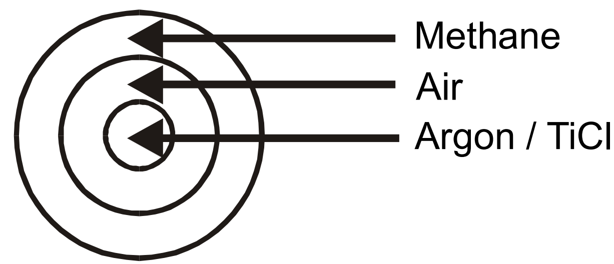


Spray flame producing Pt/Al_2O_3

Diffusion Flame Reactor for SiO_2 or TiO_2 synthesis





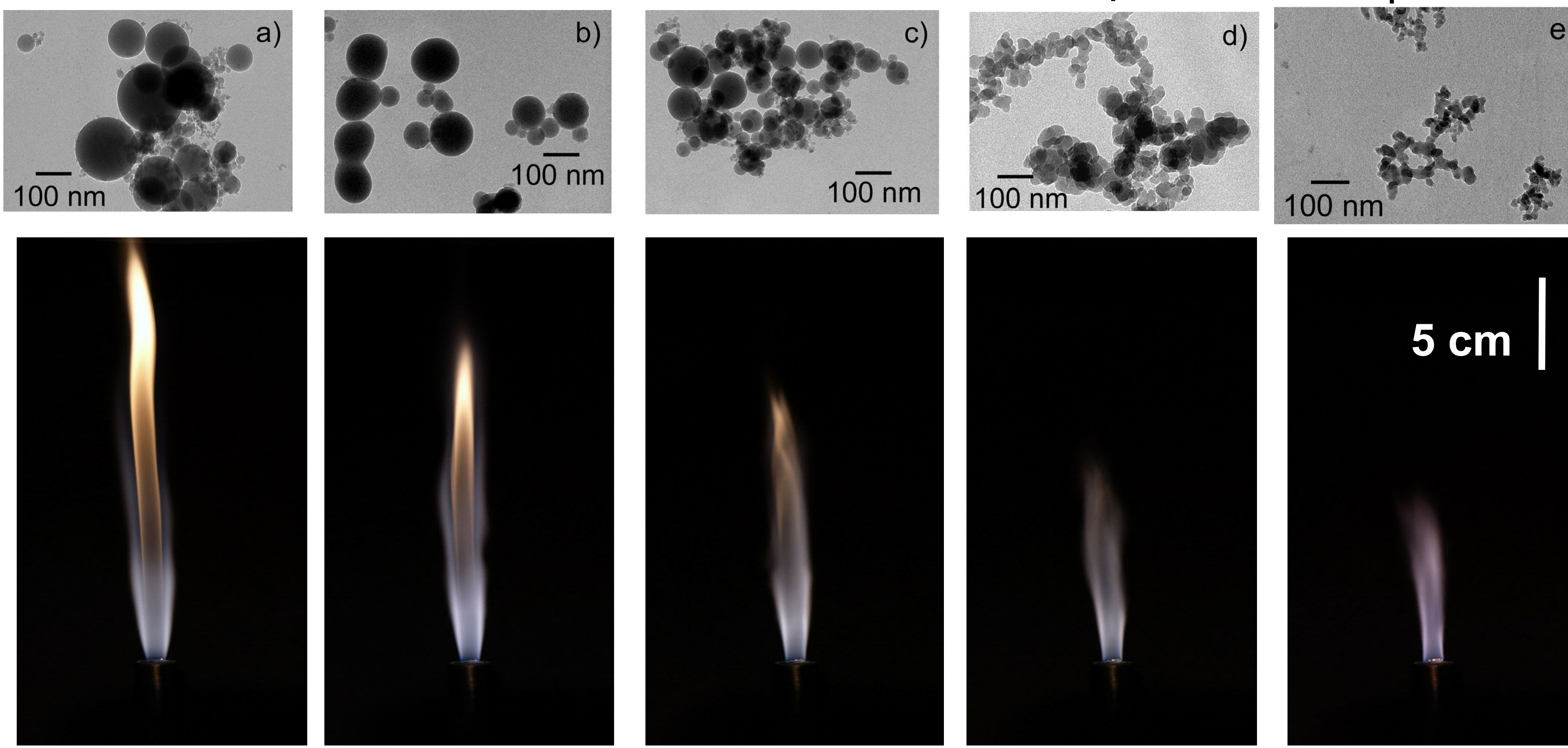


SEP, W. Zhu, S. Vemury, The role of gas mixing in flame synthesis of titania powders *Powder Technol.* **86**, 87-93 (1996).

1. Oxidant flow rate

17 g/h SiO_2

← filter powder by SAXS: non-fractal → **D_f=1.6** **D_f=1.9**

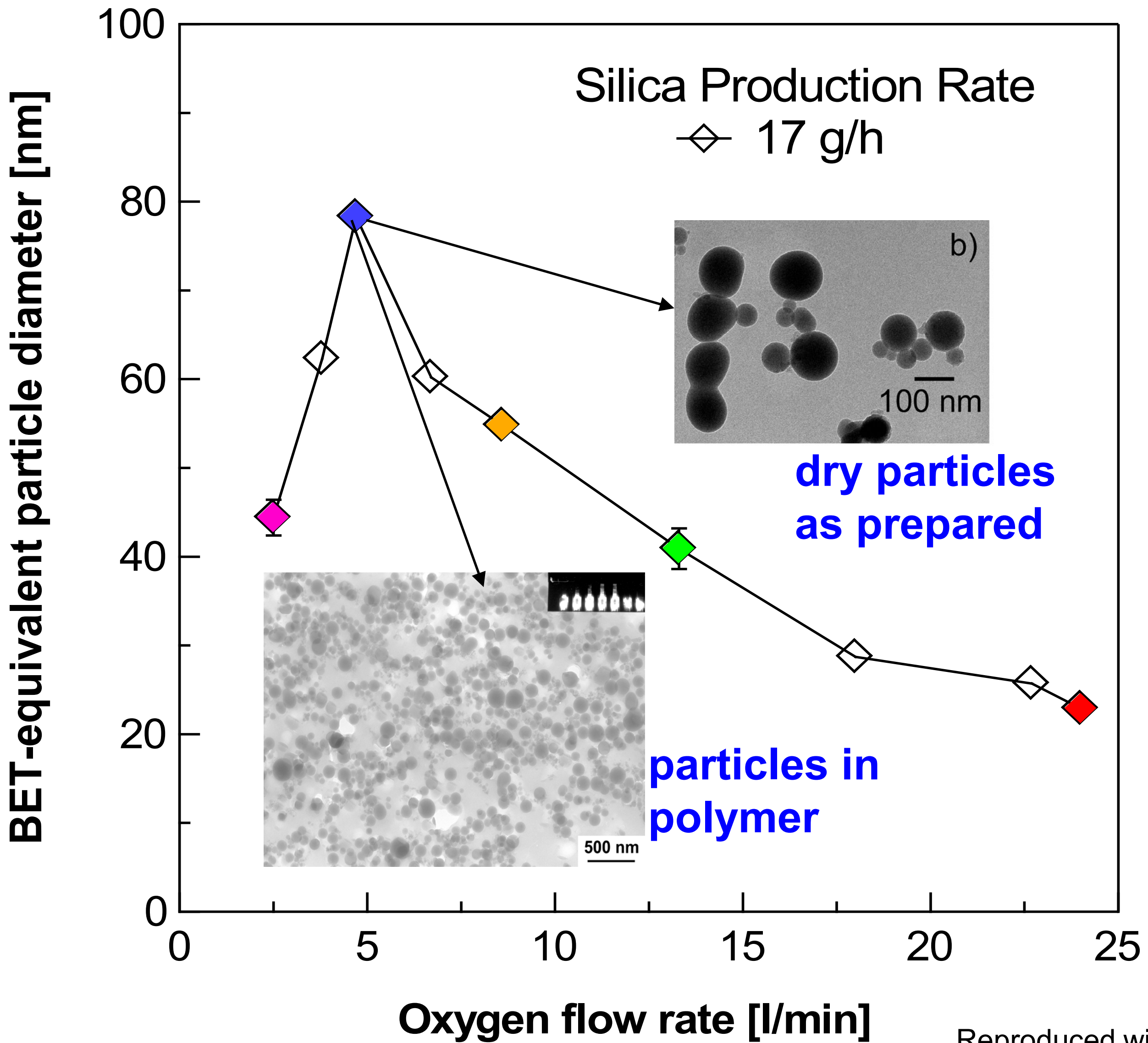


2.5 **4.7** **8.5** **13.3** **24**

Oxygen flow rate, l/min →

Mueller, Kammler, SEP, Vital, Beaucage, Burtscher, *Powder Technol.*, **140**, 40-48 (2004)

1. Oxidant flow rate



SSA = 6/ (ρ * d_p)

ρ = solid particle density

d_p = BET-equivalent particle diameter = primary particle diameter

Mueller, Kammler, SEP, Vital, Beaucage, Burtscher, *Powder Technol.*, **140**, 40-48 (2004)

1. Effect of quench (or oxidant) gas flow rate on the size (SSA) of plasma-made particles

SSA = 6/ (ρ * d_p)

GeO made in a RF plasma

SSA = 11 19 23 m^2/g

ρ = solid particle density

Scanning Electron Microscopy pictures of spherical particles with decreasing average size

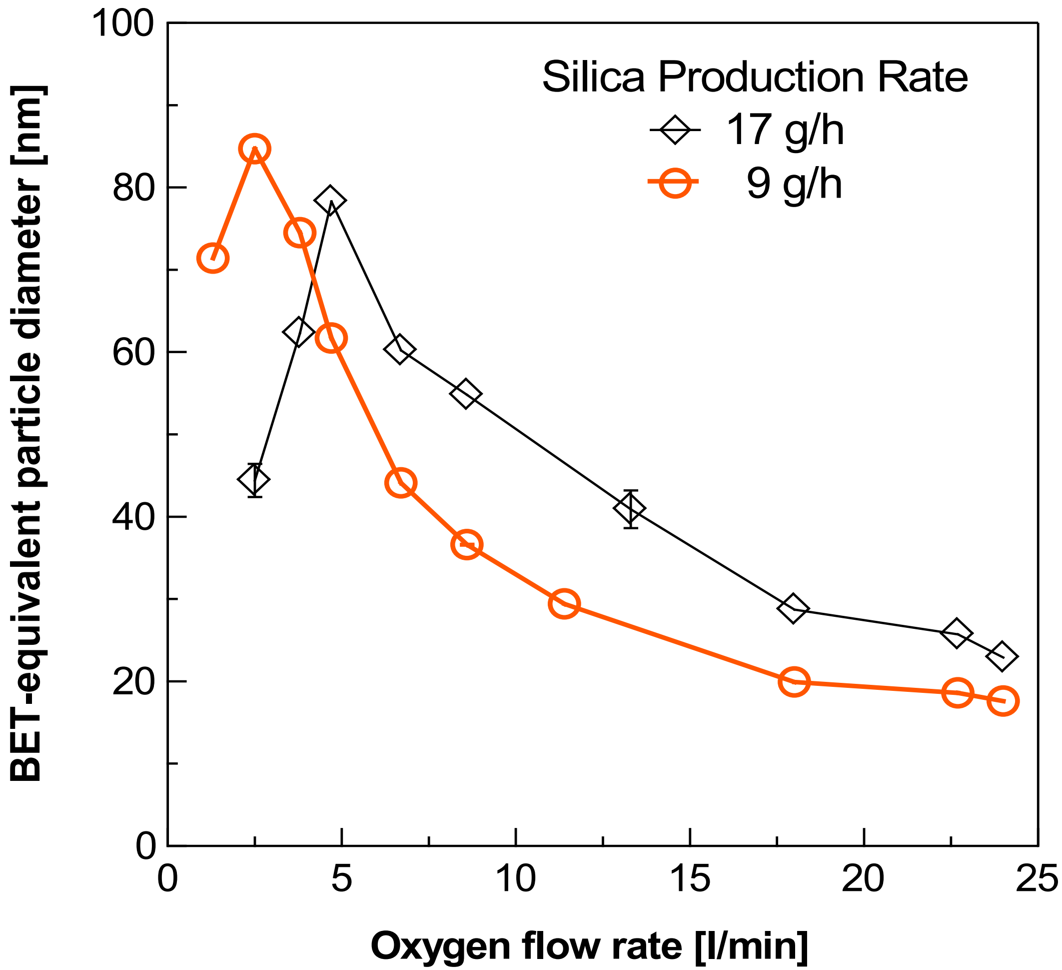
d_p = BET-equivalent (or primary) particle diameter

Quench (or Oxidant) gas flow rate

→

Vollath, D. (2008). Plasma Synthesis of nanopowders. *J. Nanopart. Res.,* **10,** 39–57. Fig 7

2. Precursor concentration



T_{HMDSO}: 5° C

Production rate 17 g/h:

- N_2 = 2.9 l/min
- CH_4 = 1.4 l/min

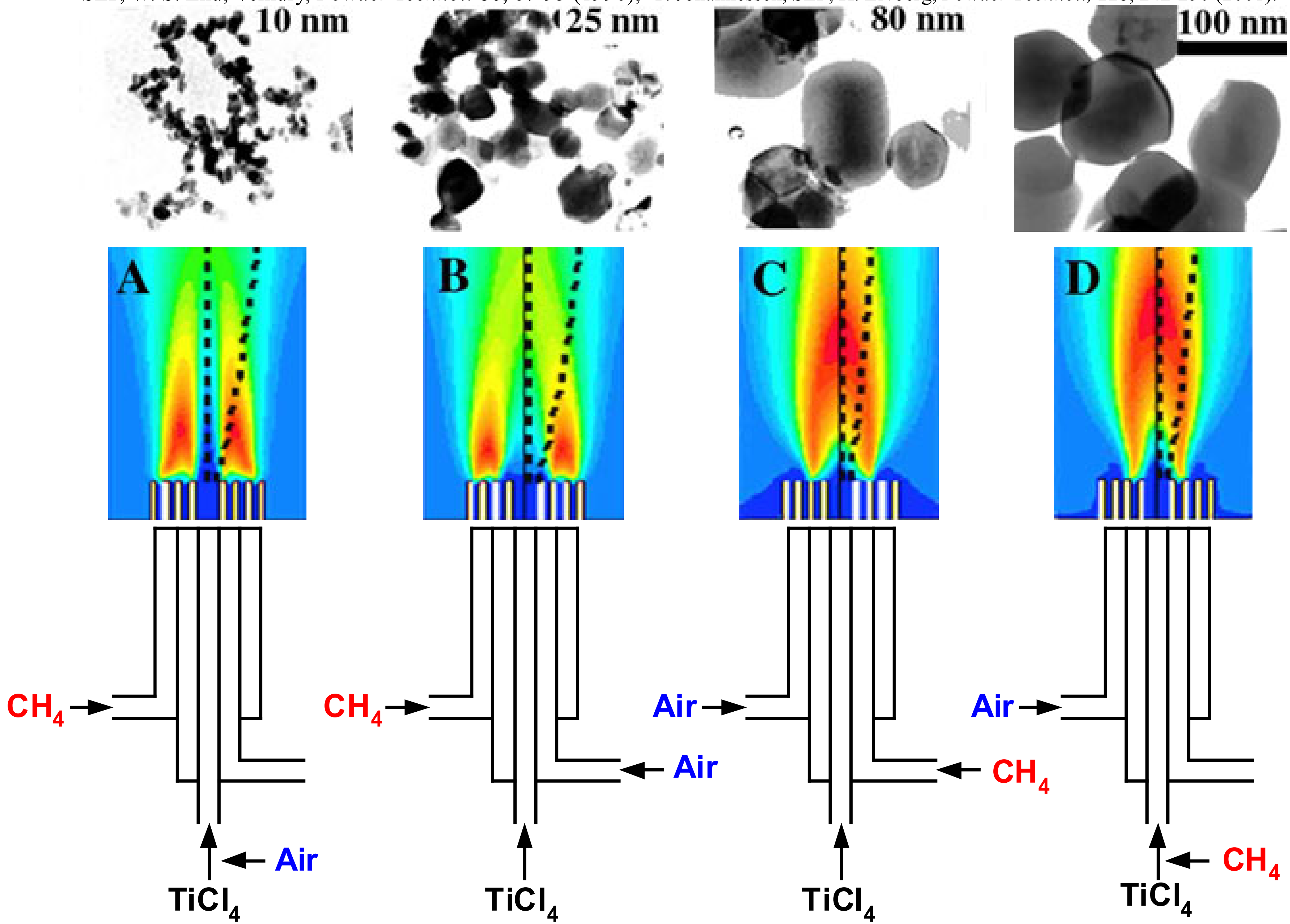
Production rate 9 g/h:

- N_2 = 1.4 l/min
- CH_4 = 0.7 l/min

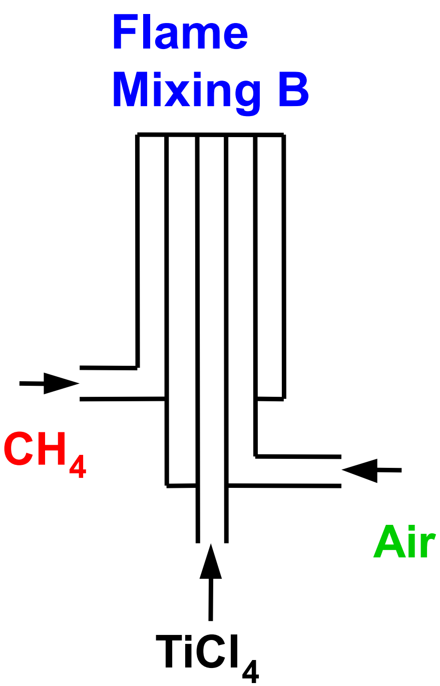
Mueller, Kammler, SEP, Vital, Beaucage, Burtscher, Non-agglomerated Dry Silica Nanoparticles *Powder Technol.*, **140**, 40-48 (2004)

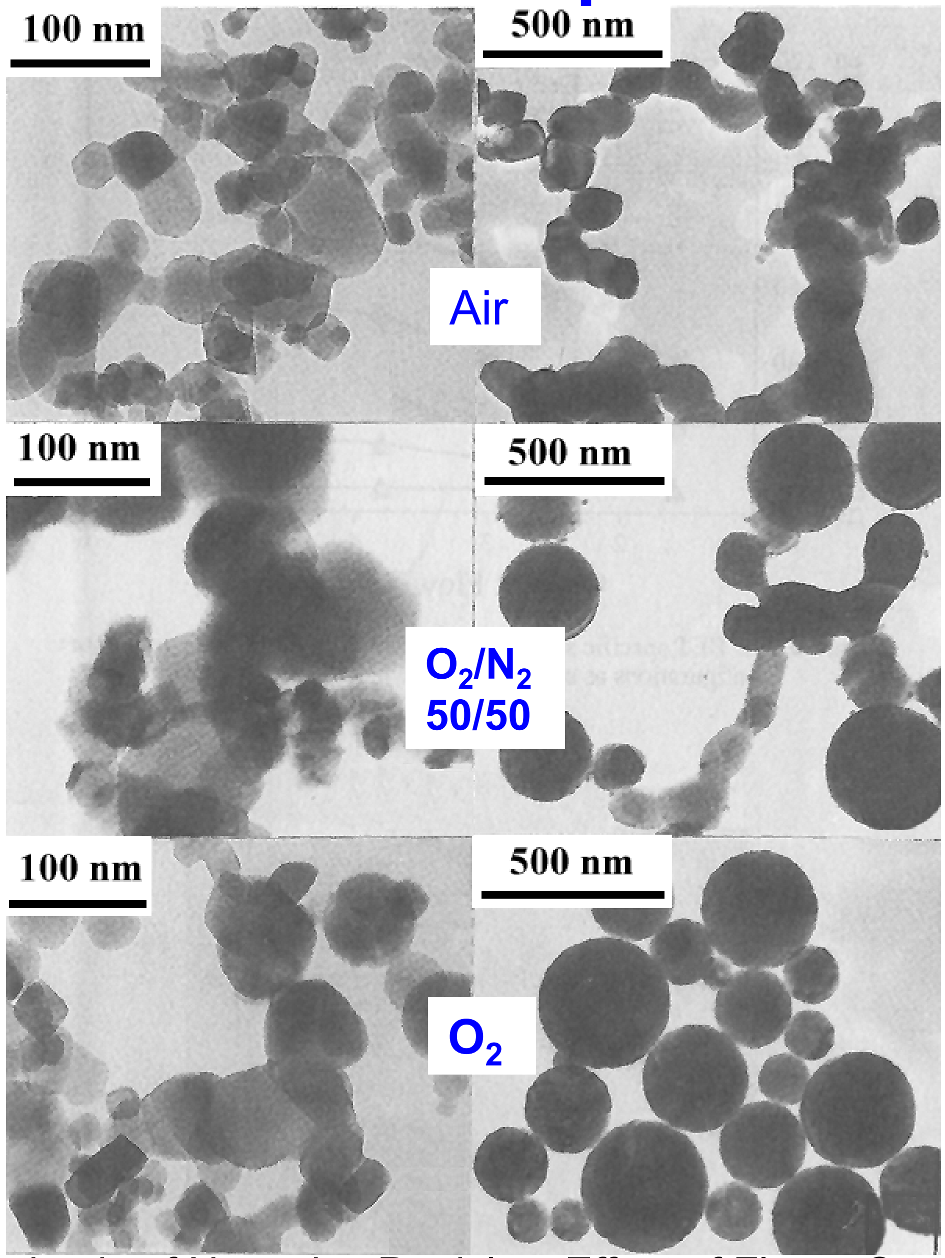
3. Mixing of reactants

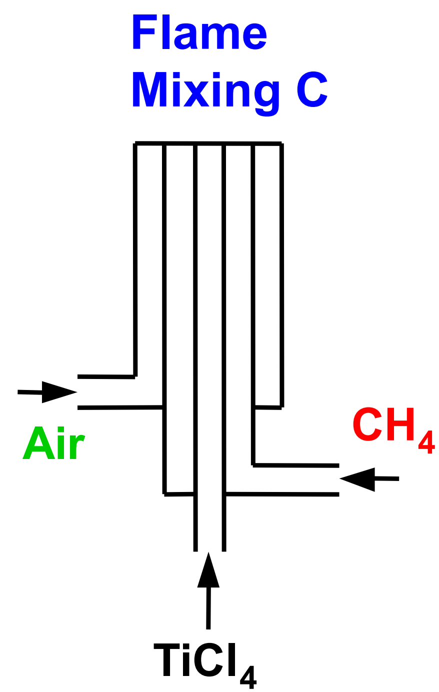
SEP, W. S. Zhu, Vemury, *Powder Technol.* **86,** 87-93 (1996); T. Johannessen, SEP, H. Livberg, *Powder Technol.,* **118**, 242-250 (2001).



4. oxidant composition

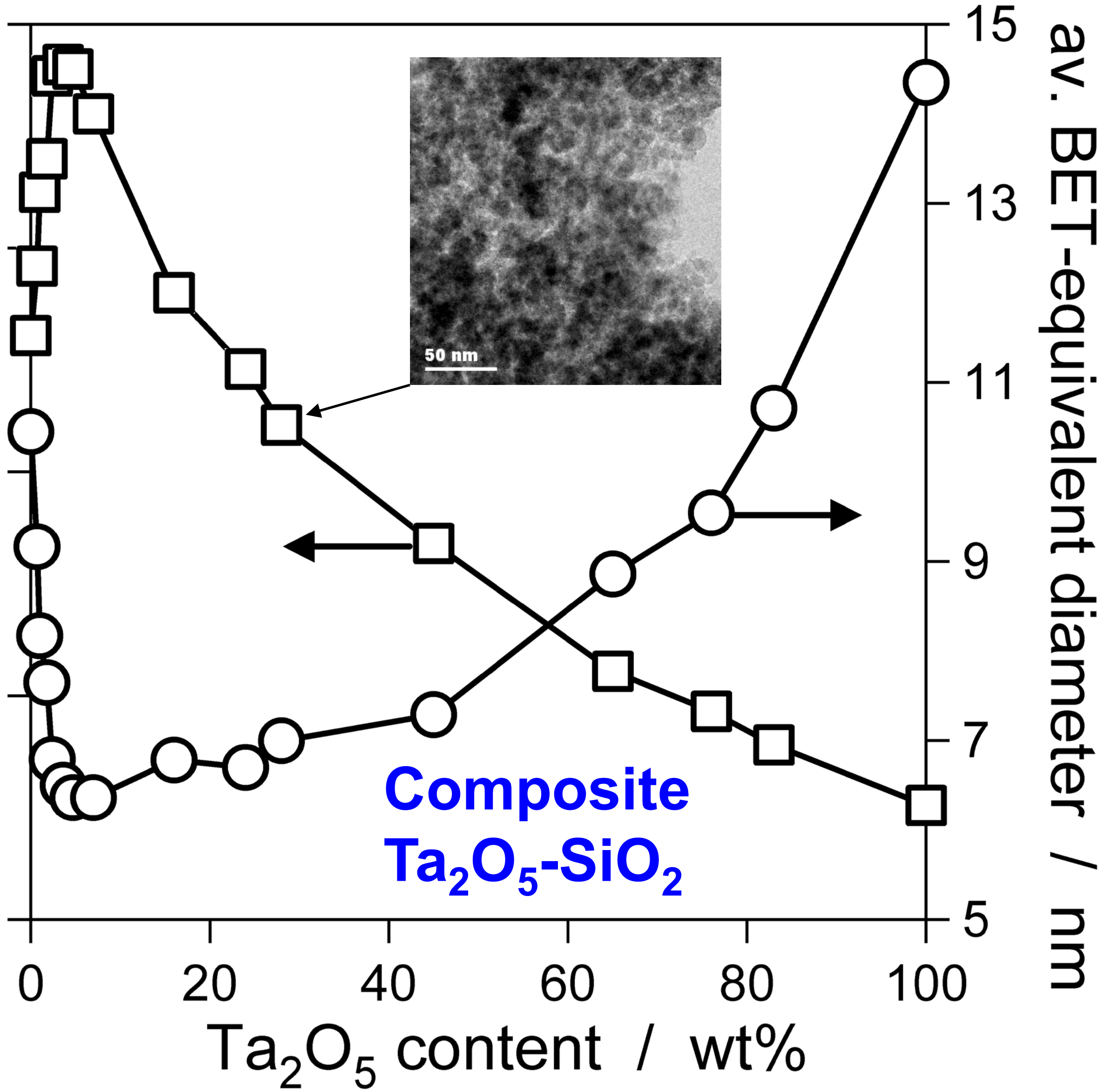


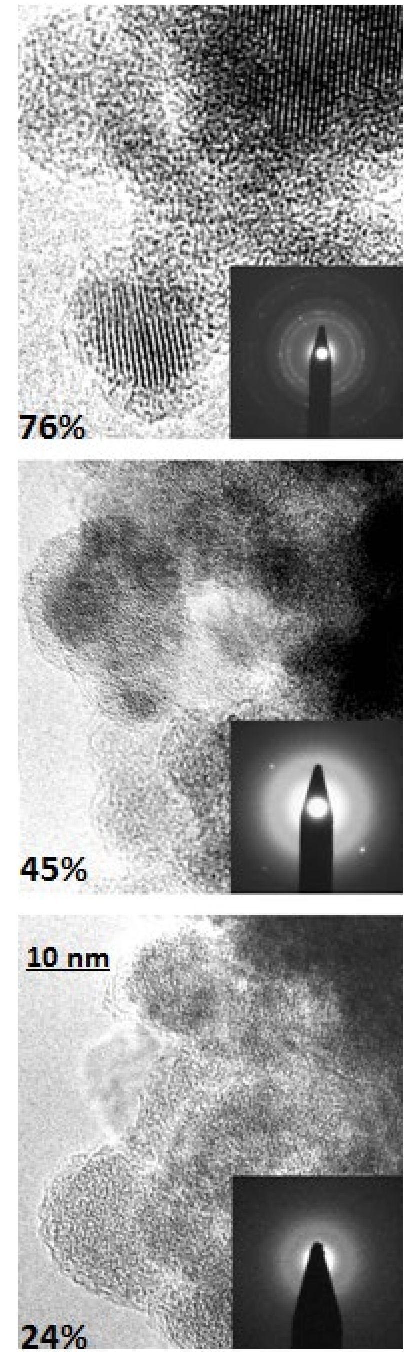


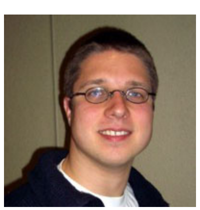


W. Zhu, SEP, Flame Synthesis of Nanosize Particles: Effect of Flame Configuration and Oxidant Composition ACS Symp. Ser. **622,** 64-78 (1996).

5. Effect of doping:Ta-content on primary particle size

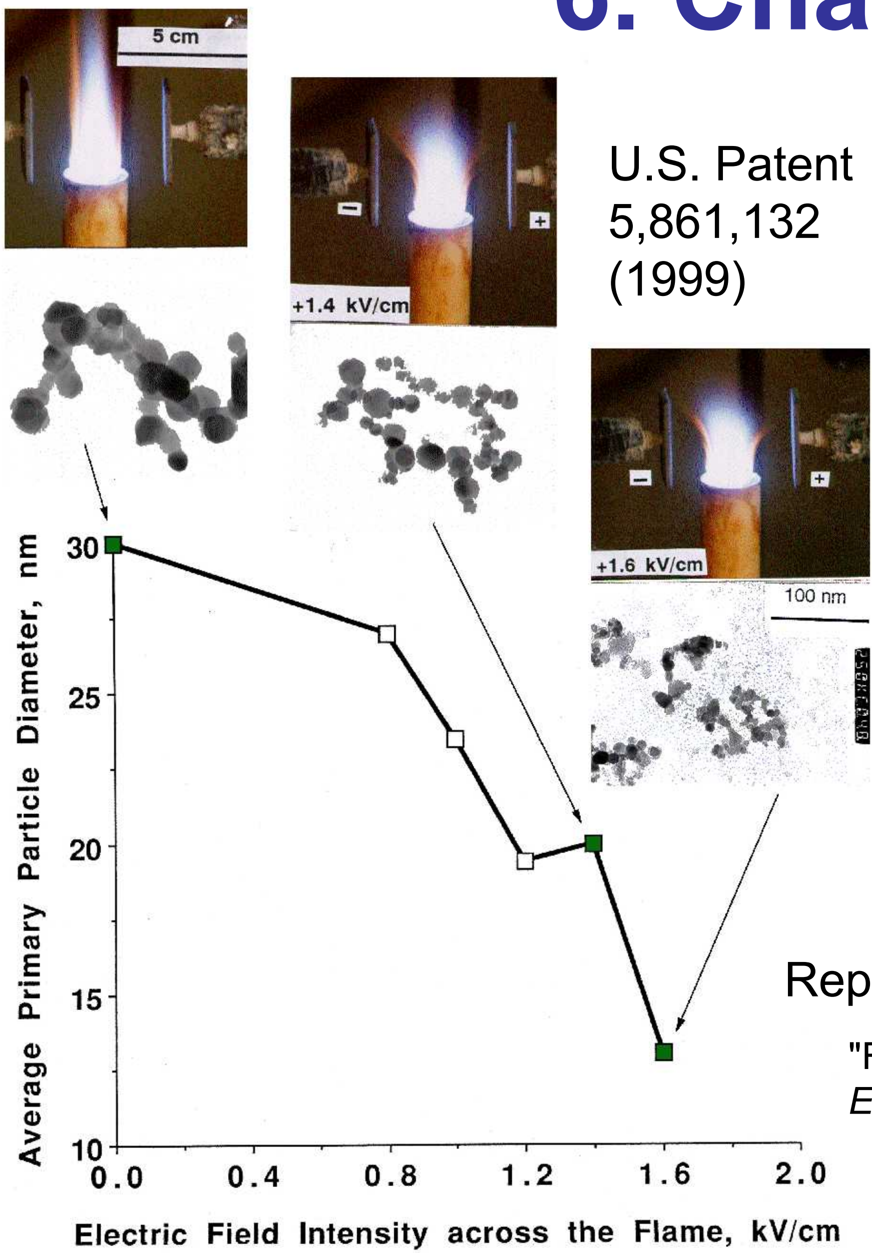




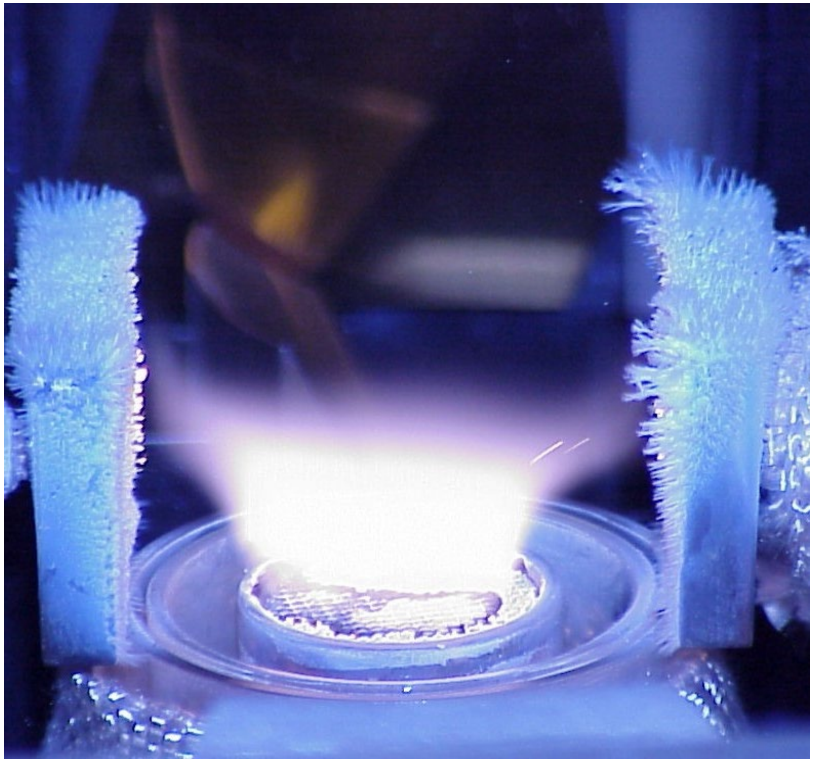


Schulz, Madler, SEP, Burtscher, Mozner, Transparent nanocomposites of radiopaque, flame-made Ta2O5/SiO2 particles in an acrylic matrix", *Adv. Functional Mater.,* **15**(5), 830-837 (2005).

6. Charging



U.S. Patent 5,861,132 (1999)



Kammler, *PhD thesis, ETH #14622* (2002)

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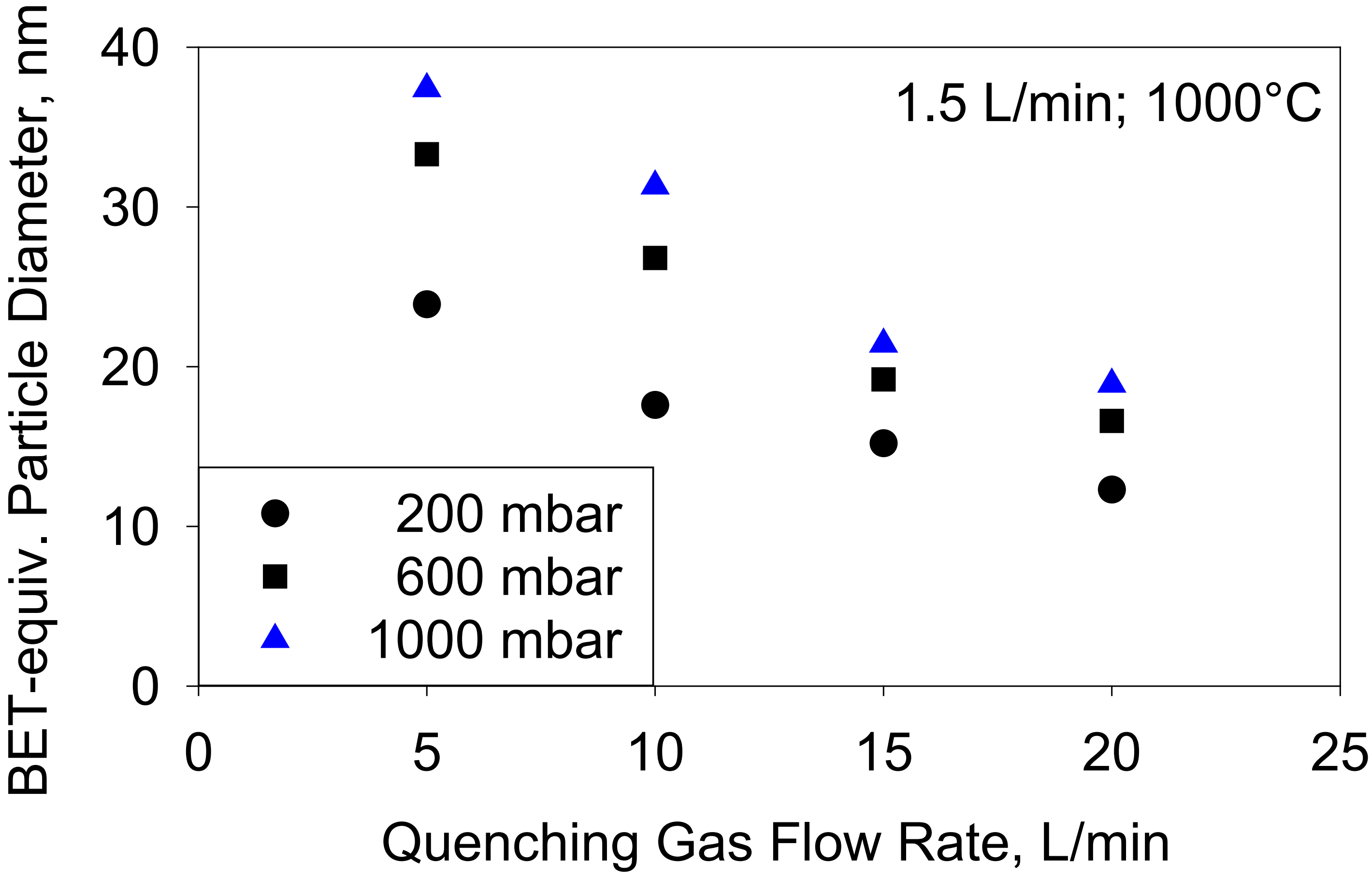
"Flame Aerosol Synthesis of Ceramic Powders" *Prog. Energy Combust. Sci.* **24**, 197-219 (1998).

S. Vemury, SEP, L. Kibbey, Electrically-Controlled Flame Synthesis of Nanophase TiO_2, SiO_2 and SnO_2 Powders *J. Mater. Res.* **12**, 1031-1042 (1997).

7a. Effect of Pressure in Aerosol (Hot –Wall) Reactors



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K. Wegner, B. Walker, S. Tsantilis, SEP, "Design of Metal Nanoparticle synthesis by Vapor Flow Condensation", *Chem. Eng. Sci.* **57**, 1753-1762 (2002).

7b. Effect of Argon Pressure in Inert Gas Condensation (Cold-Wall Aerosol Unit)

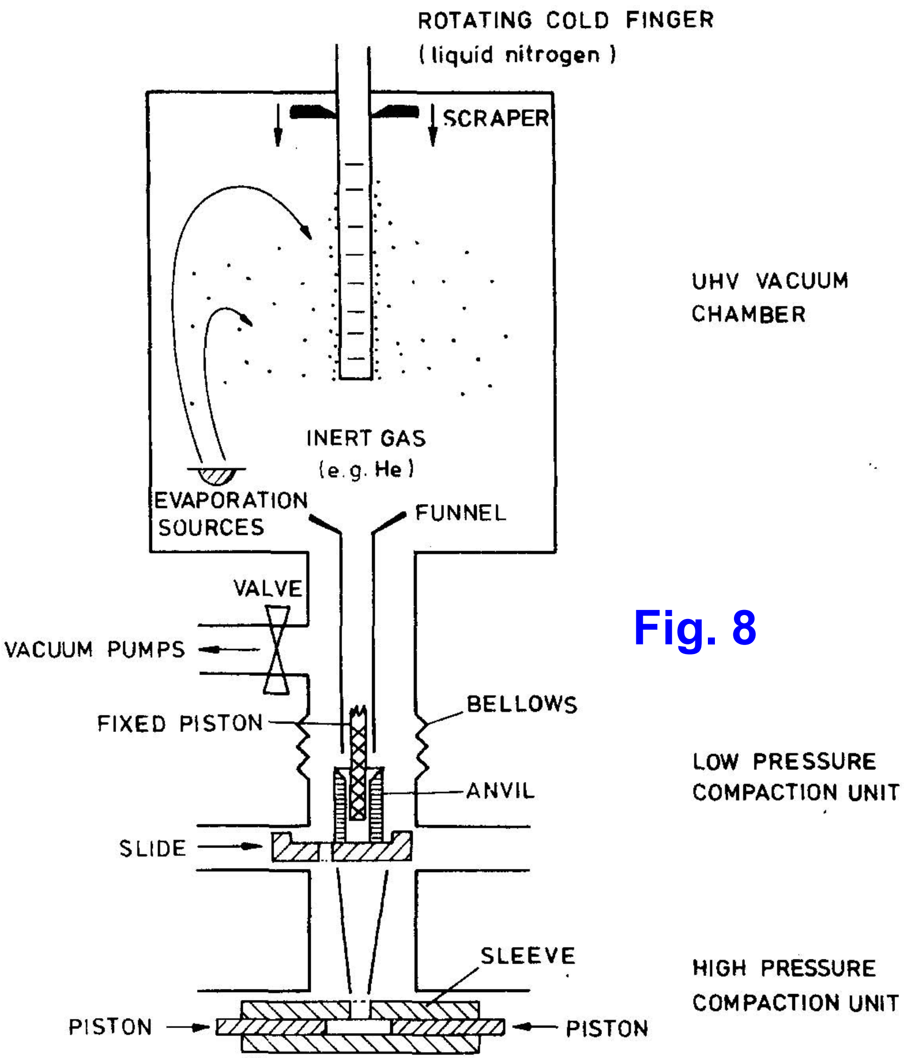


Fig. 8

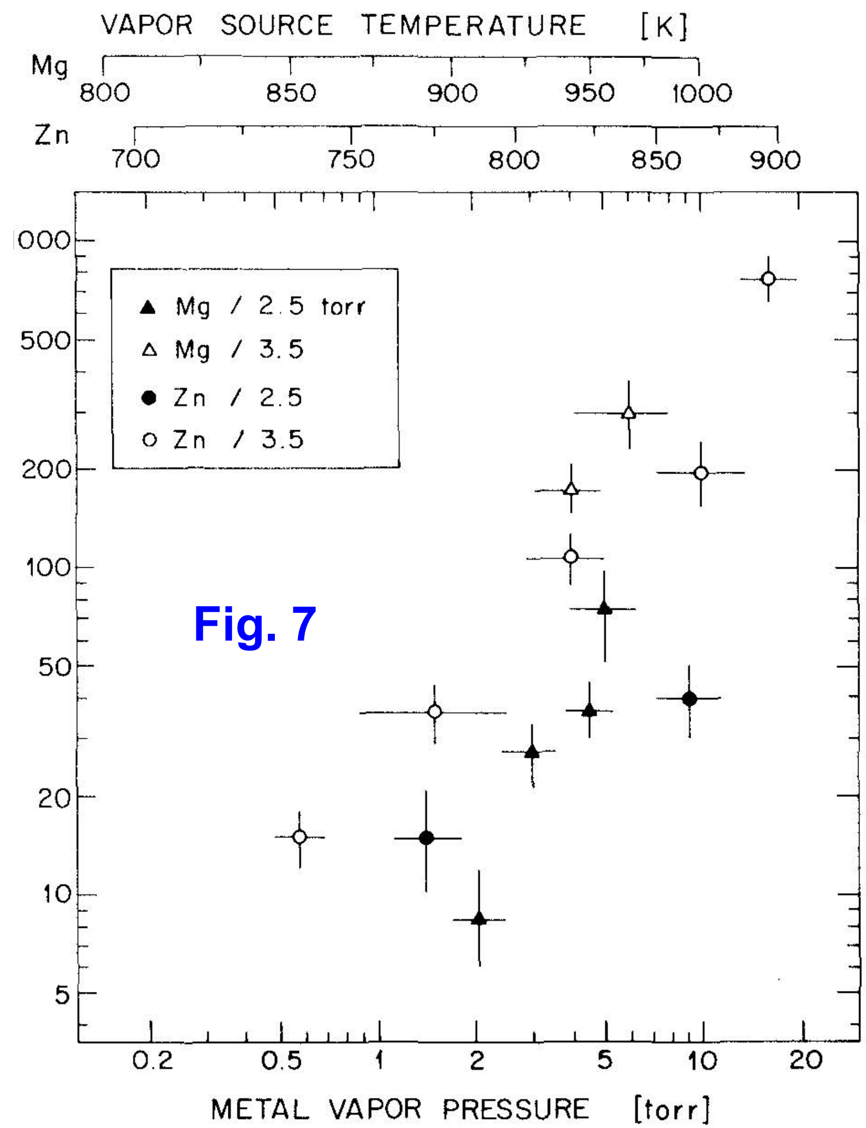
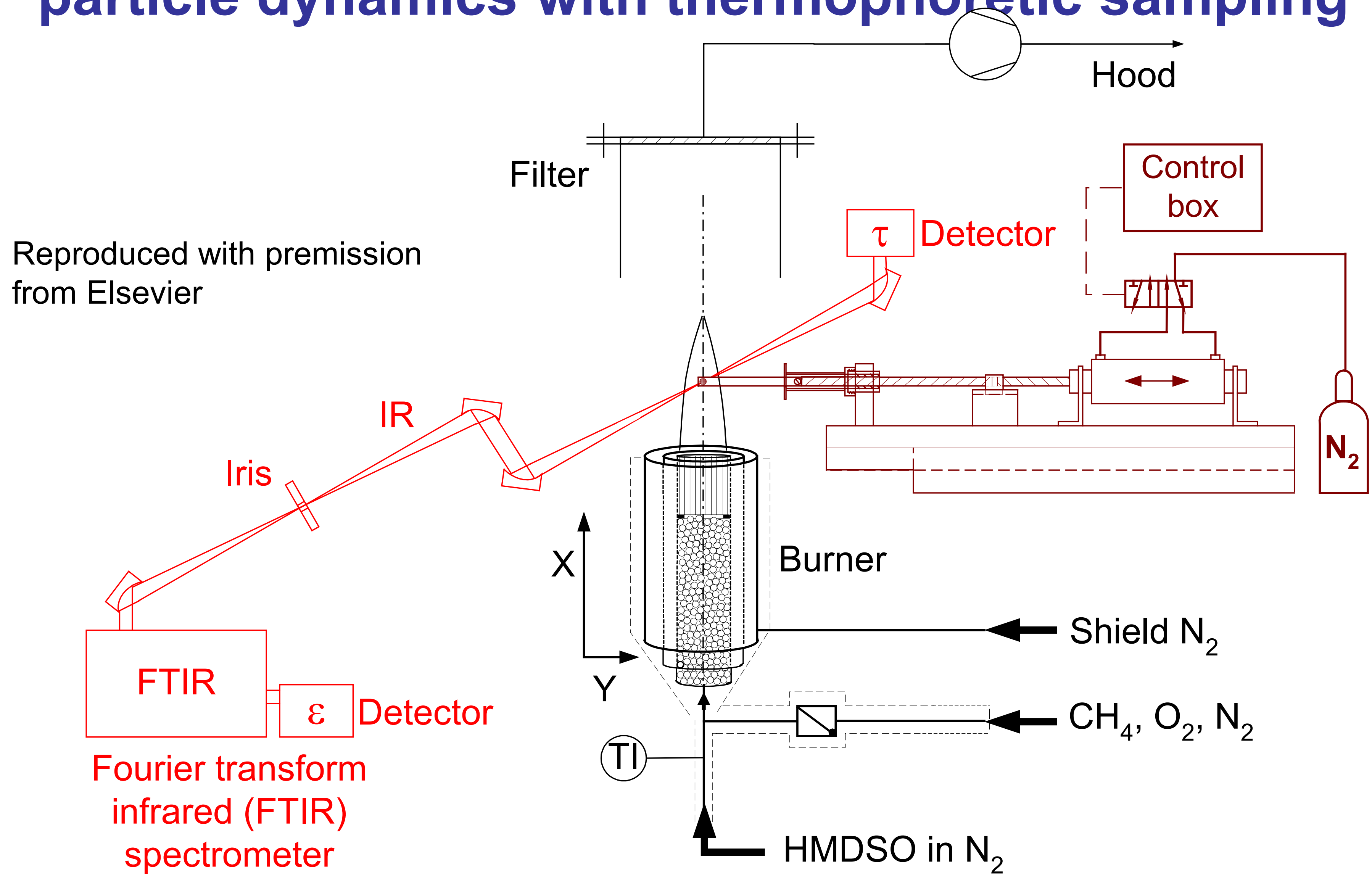


Fig. 7

Reproduced with premission from Elsevier

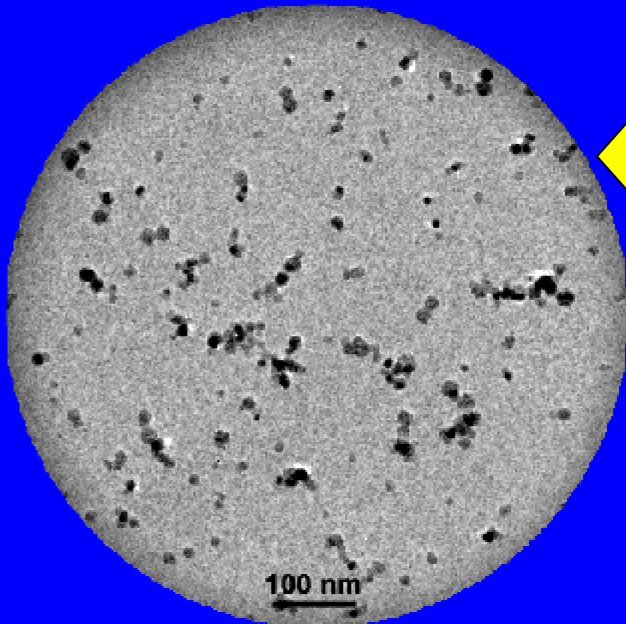
H. Gleiter, Nanocrystalline Materials, *Progress in Materials Science*, **33**, 223-315 (1989)
C.G. Granqvist, R.A. Buhrman, Ultrafine Metal Particles, *J. Appl. Physics*, **47**, 2200-2219 (1976) Fig. 7

Open the “black box” of the flame by monitoring particle dynamics with thermophoretic sampling



Kammler, R. Jossen, P.W. Morrison Jr., S.E. Pratsinis, G. Beaucage "The effect of external electric fields during flame synthesis of titania", *Powder Technol.*, **135**:310 (2003).

Thermophoretic Sampling (Dobbins & Megaridis, Langmuir 3, 254, 1987)



TEM

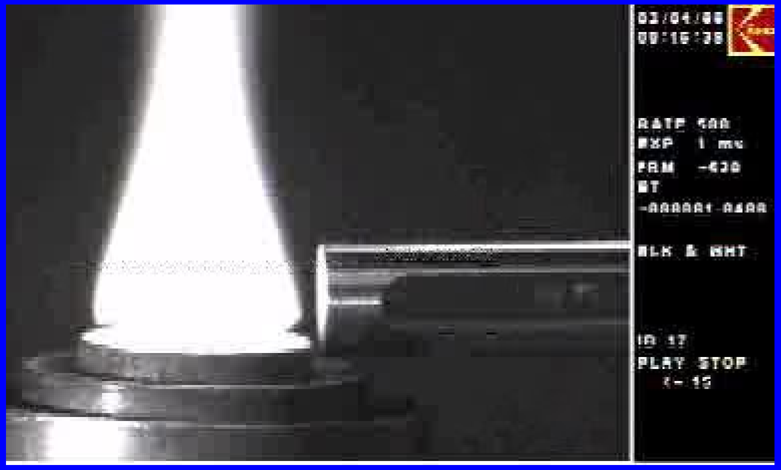
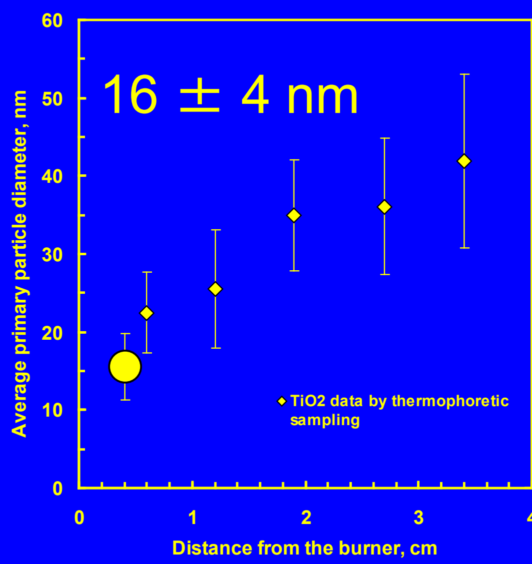


Image Analysis



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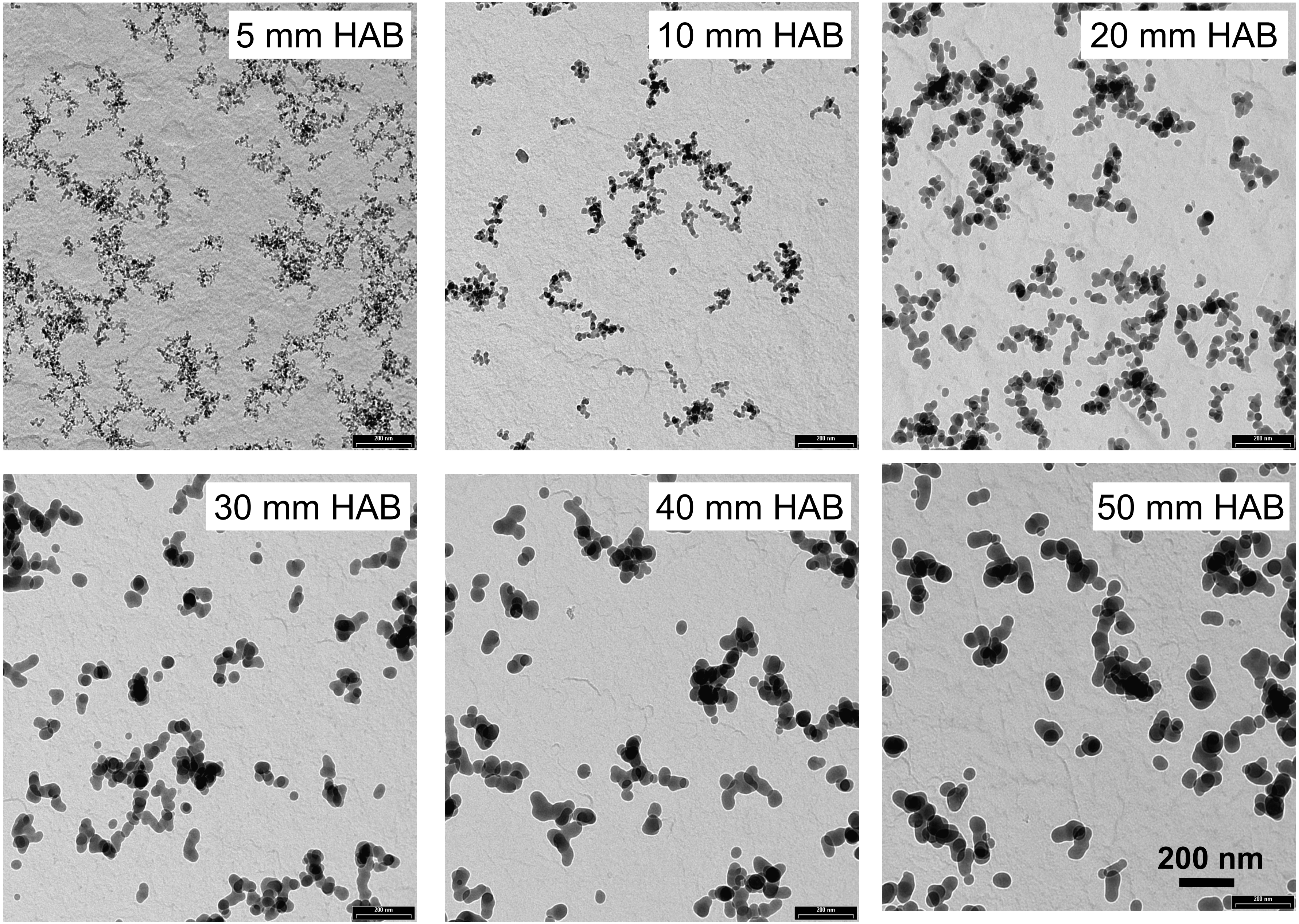
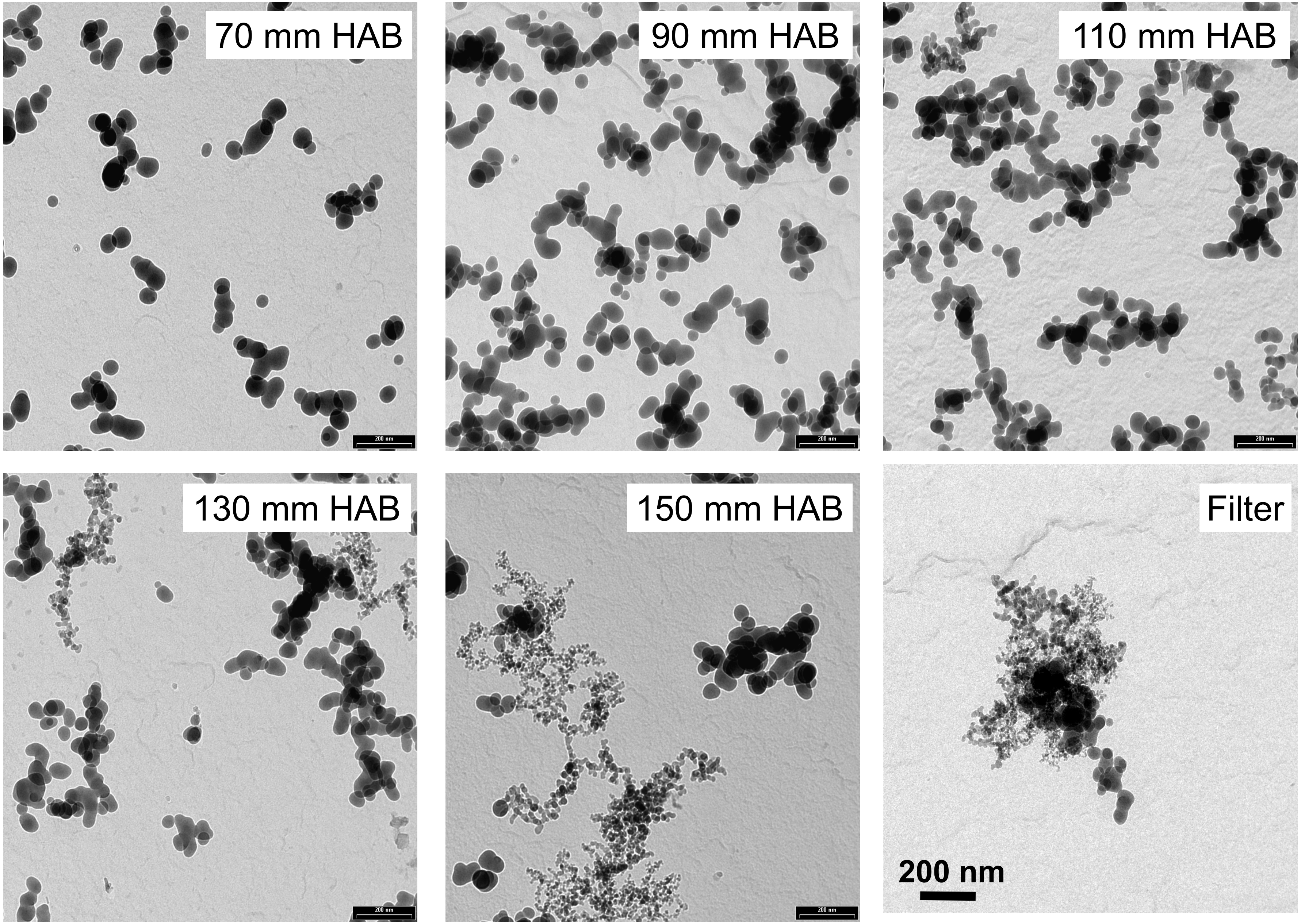
S. Tsantilis, H.K. Kammler and Pratsinis, S.E. "Population Balance Modeling of Flame Synthesis of Nanoparticles", *Chem. Eng. Sci.*, **57**, 2139-56 (2002).

Axial distribution of flame-made SiO_2 nanoparticles: Part 1



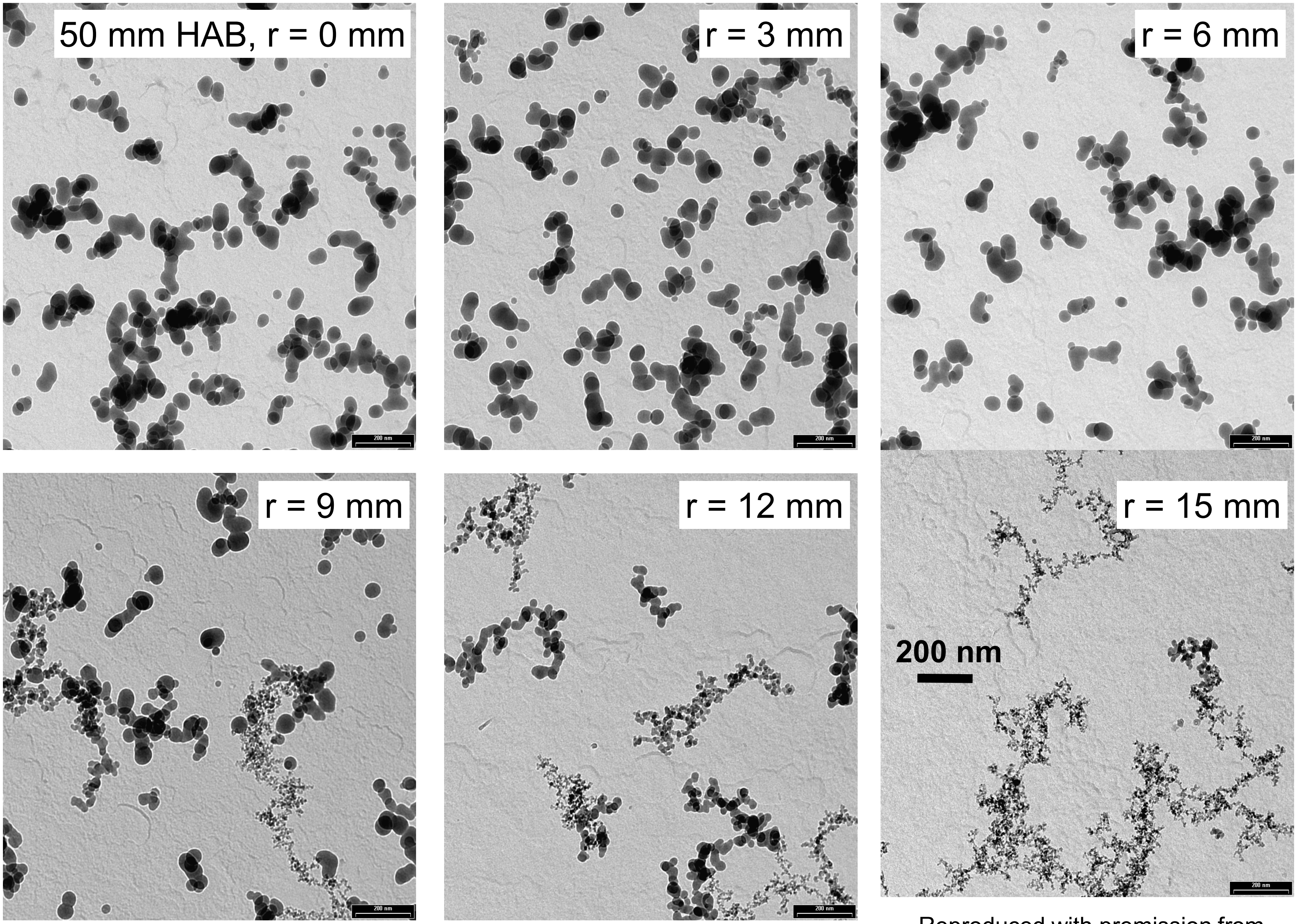
H.K. Kammler G. Beaucage et al., *J. Appl. Phys*., **97**, 054309 (2005).

Axial distribution of flame-made SiO_2 nanoparticles: Part 2



H.K. Kammler G. Beaucage et al., *J. Appl. Phys.*, **97**, 054309 (2005).

Radial distribution of flame-made SiO_2 nanoparticles



H.K. Kammler G. Beaucage et al., *J. Appl. Phys*., **97**, 054309 (2005).

Conclusions

The size, crystallinity and morphology of simple oxide nanoparticles made in flames can be controlled through a number of process variables, including but not limited to:

1. Oxidant flow (temperature, aerosol dilution)
2. Precursor/fuel composition and concentration
3. Oxidant composition (temperature history)
4. Mixing of oxidant and precursor (temperature history)
5. Doping (material properties→ coalescence)
6. External (e.g. electrical) fields (charging & temperature)
7. Pressure (particle collision frequency & coagualtion)

Thermophoretic sampling offers a glimpse on particle dynamics