Synthesis of Non-Aggregated Silica Nanoparticles

*T. Graule, *A. Vital, *U. Klotz
§S.E. Pratsinis, §R. Müller, §H. Kammler
°N. Moszner, °P. Burtscher

Objectives

ETHZ and EMPA have investigated the synthesis of spherical, non-agglomerated silica (SiO_2) nanoparticles with a mean diameter <100nm by vapour flame aerosol technology. Commercially available silica nanoparticles, have a nominal primary particle diameter of 7-70 nm. However, they are highly aggregated and form clusters of 200-300 nm in size (Fig.1).

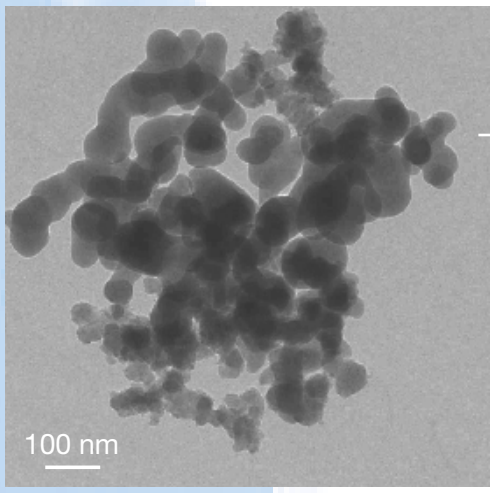


Figure 1 - Commercial SiO_2 nanopowder (Degussa, Aerosil OX50, d_{50}=55 nm) forming large aggregates

By application of the spherical, non-aggregated SiO_2 nanoparticles (Fig. 2) as filler in polymer/ceramic composites, significant improvements of the optical and mechanical characteristics of the composite were expected due to an enhanced homogeneity of the filler material within the polymer matrix.

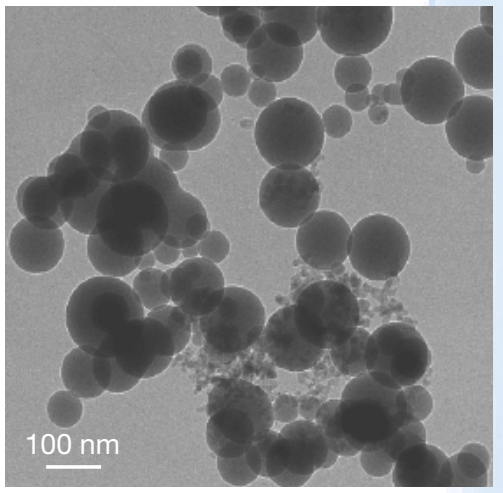
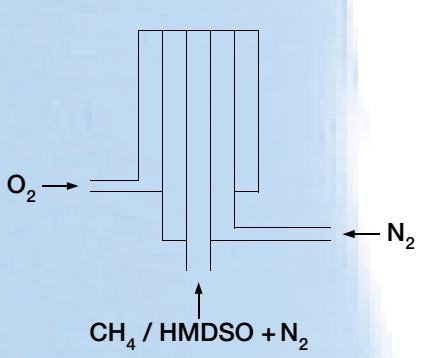


Figure 2 - Non-aggregated, spherical SiO_2 nanopowder (d_{50} =77 nm) synthesized via flame aerosol technology by ETHZ / EMPA.

Experimental Procedure

A lab-scale aerosol flame reactor designed at ETHZ was used for particle synthesis. The modular plant set-up and burner design, consisting of three concentric gas outlet tubes (Fig. 3, 4), allowed exact control of the flame configuration and process parameters such as precursor feeding rate, gas flow rates and powder production rate.



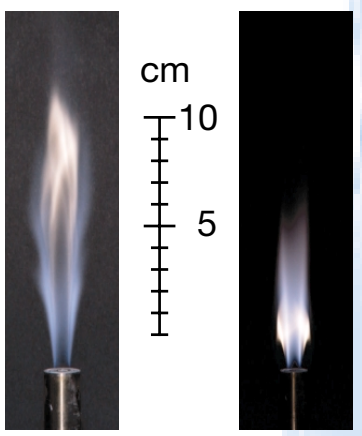


Figure 3 - Schematic and appearance of a single diffusion flame (SDF)

Hexamethyldisiloxane (HMDSO) was used as precursor and evaporated from a bubbler flask at a constant temperature. Nitrogen was supplied as inert carrier gas and methane was provided as fuel gas.
The particles were characterized by BET method for determination of a mean, equivalent particle diameter and TEM was applied for morphological examination.

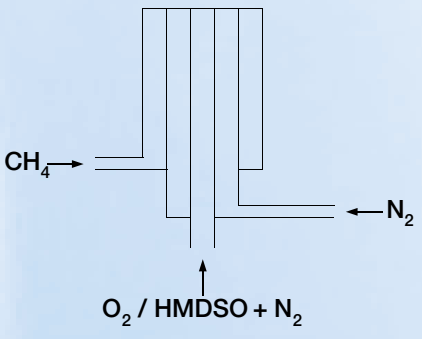


Figure 4 - Schematic and appearance of a double diffusion flame (DDF)

Results and Conclusion

Reproducible synthesis of non-aggregated SiO_2 nanoparticles with a mean diameter of <100nm has been attained. A systematic parameter investigation revealed that powder with a primary particle-diameter of 10 - 90nm can be synthesized (Fig 5). The particles show various morphologies from a non-aggregated, spherical appearance (Fig. 6a) to a highly-aggregated, fractal-like structure (Fig. 6b).

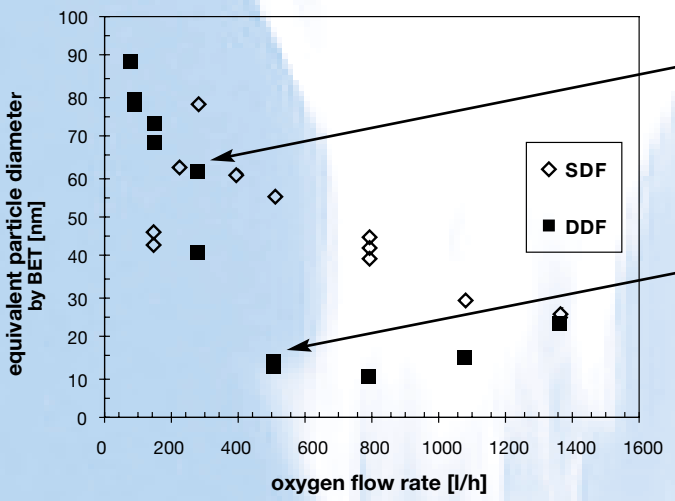


Figure 5 - Correlation of mean particle diameter with oxygen flow rate for a single diffusion flame (SDF) and a double diffusion flame (DDF)

With the non-aggregated SiO_2 particles (Fig. 6a), Ivoclar has manufactured ceramic/polymer composites and determined their properties. In comparison to composites prepared of commercial silica nanoparticles (Aerosil OX50), the composites show an extraordinary opalescence effect and significantly improved mechanical characteristics, such as Vicker's hardness (+20%), flexural strength (+10%) and flexural modulus (+13%).

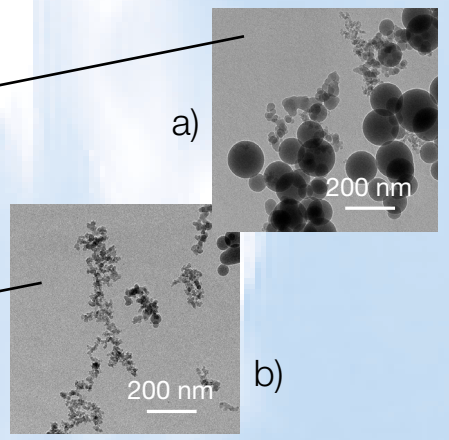


Figure 6 - TEM micrographs of a) non-aggregated, spherical particles and b) fractal-like particles

Outlook

Future projects aim upon synthesis of non-aggregated, silica-based mixed-oxide nanoparticles with a high refractive index, resulting in a ceramic/polymer composite with a dentine-like colour, high abrasion and wear resistance as well as high radiopacity.

***Swiss Federal Laboratories for Materials Testing and Research**
CH-8600 Dübendorf
Dr. A. Vital
phone: +41 (0)1 823 43 66
mail: andri.vital@empa.ch

§Swiss Federal Institute of Technology, Institute of Process Engineering,
CH-8092 Zürich
Prof. S.E. Pratsinis
phone: +41 (0)1 632 31 80
mail: pratsinis@ivuk.mavt.ethz.ch

°Ivoclar-Vivadent AG
Bendererstr. 2
FL-9494 Schaan
Prof. N. Moszner
phone: +42 3 235 35 35
mail: norbert.moszner@ivoclar.com

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