

## In-situ Small Angle X-Ray Scattering (SAXS) Characterization of SiO<sub>2</sub> Nanoparticles Synthesized in a Microwave-Plasma Reactor

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Optical, mechanical, thermal or handling properties of coatings as well as bulk materials can be improved by using nanomaterials and nanocomposites containing nanoparticles. Depending on the application, specific properties of the nanomaterials are required. For example if a high transparency of coatings with high mechanical and chemical resistance is demanded, SiO<sub>2</sub> nanoparticles with a specific size, morphology and surface coating are particularly suitable. Thus, a detailed characterization of the nanoparticles and also an exact knowledge concerning the particle formation and particle growth is indispensable.

An adequate measuring method is the small-angle X-ray scattering (SAXS), which can be used for in-situ measurements of aerosols as well as suspensions. The technique can determine the particle size and size distribution, the specific surface area, the fractal dimension and the aggregate number within one measurement (Beaucage *et al.*, 2004). Besides the multiplicity of particle describing parameters within a single measurement, the technique offers the opportunity to measure in-situ.

However, laboratory X-ray scattering equipment like Kratky and pinhole cameras has a low scattering intensity and as a consequence, the measurement takes several hours (8h+). To overcome this constraint a Kratky compact camera was modified using a multilayer X-ray mirror (Göbel mirror). The mirror converts a divergent, incoming X-ray beam into a parallel one and was established inside the camera. Additionally, a two-dimensional imaging plate detector replaced the one-dimensional, gas-filled detector. Owing to these changes, intensity and image quality are increased and the required measurement time is decreased by a factor of 20. Due to this, the camera can be used now for in-situ measurements during particle formation.

In order to validate the in-situ measurement technique the synthesis of SiO<sub>2</sub> nanoparticles in a microwave-plasma reactor is observed. Inside the plasma a gas mixture including TEOS vapour, oxygen, argon, and nitrogen is decomposed within a few microseconds followed by the formation of silica nanoparticles.

With respect to the reactor design, the SAXS camera body is split in two parts to integrate the reaction chamber into the measurement section and

to operate the camera in the particle formation zone. In Figure 1 the in-situ measurement setup is represented.

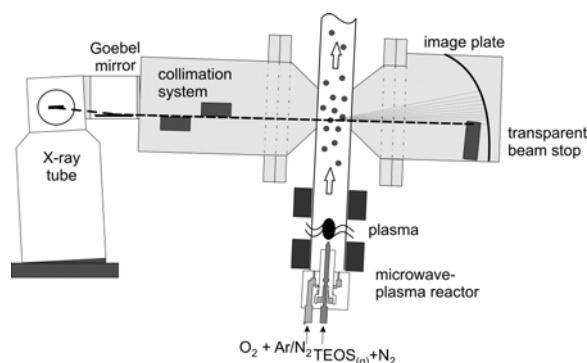


Figure 1. Schematic representation of the in-situ SAXS measurement setup

The in-situ SAXS results are compared with online particle mass spectrometry and TEM measurements (Janzen *et al.*, 2001). For the TEM measurements, particles collected from the PMS molecular beam and through thermophoretic sampling of nanoparticles from the reaction chamber were investigated. The results show a good match between the different measurement techniques and qualify the SAXS method as a non-intrusive possibility for the in-situ characterization of nanoparticle properties.

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