On-line Diagnostics for Particle Size in Nanoparticle Manufacturing

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Introduction

Real-time aerosol characterization is not only important for airborne pollutants but also for monitoring of continuous aerosol processes, such as the production of engineered nanoparticles. For instance, the primary particle and agglomerate sizes often govern particle properties and must be controlled in a narrow range in order to achieve certain product performance. Flame aerosol reactors that dominate the field in terms of throughput, cost and versatility have production rates up to 1 ton per hour. Here, real-time online diagnostics for particle size can assist process control, assuring production quality and resulting in significant cost savings.

Sampling these production streams, however, is all but trivial. Particle concentrations are as high as 10^{18} #/kg_{gas}, requiring an enormous dilution of the aerosol to quench particle growth processes. The probe has to withstand temperatures up to ~1500 K and possibly corrosive environments. For this reason, mostly ex-situ methods have been used to determine particle size and morphology.

Here, an in-situ method for real-time determination of average agglomerate mass, volume, mobility and structure along with the constituent primary particle size is presented. It is performance-tested in continuous lab- and pilot-scale production lines for nanoparticles made by flame spray pyrolysis (FSP; Mädler et al., 2002).

Method

Flame synthesis of zirconia nanoparticles at rates up to 2 kg/h is used as a test system. A sampling probe for continuous extraction of the hot and highly concentrated aerosol was designed and constructed. Immediate dilution with adjustable cooling air flow rate allows effective suppression of coagulation. This enables in addition to continuous and real-time process monitoring direct measurement of the primary and agglomerate particle growth dynamics.

Online characterization of the fractal-like particles is achieved by combining a differential mobility analyser (DMA) with an aerosol particle mass analyser (APM) and applying a power-law correlation to derive average primary particle size as proposed by Eggersdorfer et al. (2012).

Results of the in-situ measurements are compared against off-line particle size characterization as well as predictions of a computational fluid dynamics (CFD)-based process model interfacing spray, fluid, combustion and aerosol dynamics (Gröhn et al., 2011, 2012).

Results and Discussion

Figure 1 presents how process simulations (Gröhn et al., 2012) show an increase in the agglomerate size and degree of aggregation for increasing precursor concentration consistent with experimental data at 4 ml/min precursor feed and dispersion O_2 flow of 5 l/min. Also the onset of soft-agglomerate formation can be determined from simulations. More detailed sampling at different radial and axial positions in the FSP reactor are to be conducted for improved model validation. Already good quantitative agreement is attained between the filter samples, CFD predictions and on-line sampling.

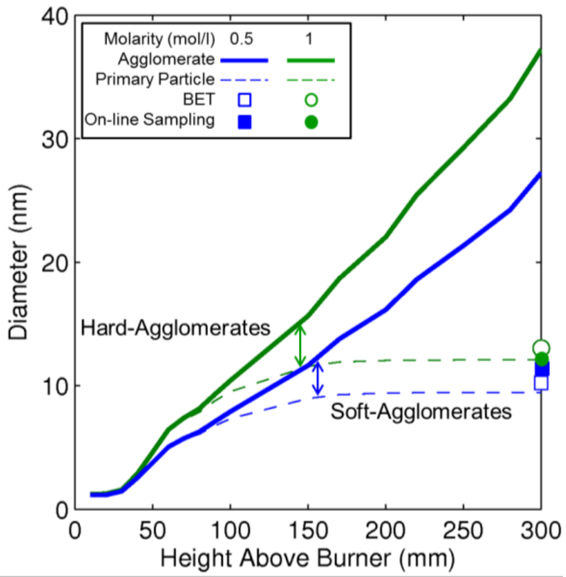

Figure 1. Predicted evolution of primary particle and agglomerate collision diameters for 4 ml/min of 0.5 and 1 M Zr precursor. Initiation of soft-agglomerate formation is indicated with arrows.

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