Agglomerate Stability in Orifices

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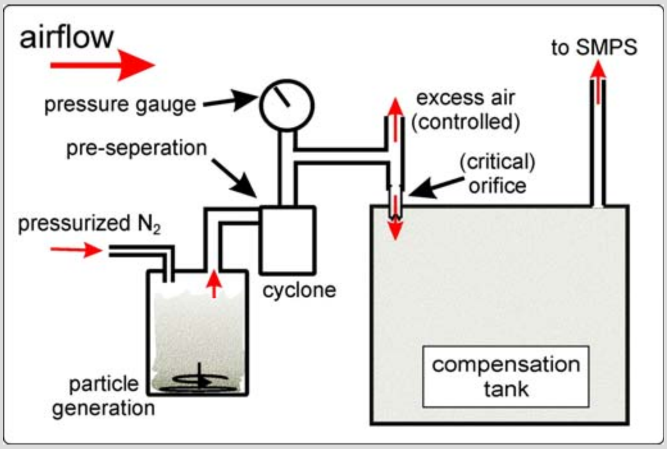
Abstract

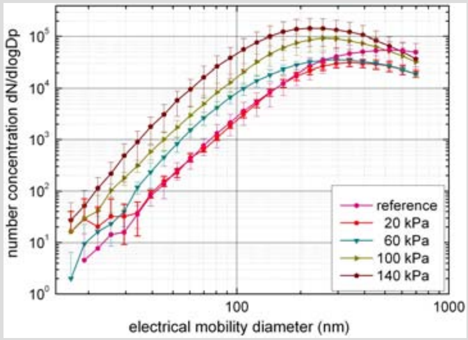
During handling of agglomerated nanopowders, agglomerates may break up into smaller aggregates (tightly bound) or even primary particles when the effect of applied shear forces exceeds the binding energy. To study the stability of agglomerates we performed measurements of aerosolized nanopowders passing a (critical) orifice. The aerosol is prepared in a beaker under different pressure conditions ranging from 20 kPa up to 140 kPa above ambient pressure. After generation the aerosol passes an orifice and the resulting particle size distribution is measured by a scanning mobility particle sizer. Due to shear forces within the orifice agglomerates may break up when passing the orifice. The obtained size distribution is compared to that measured directly after generation, i.e. without passing the orifice. This allows the calculation of relative increase or decrease of particle number for a given mobility diameter. We present results for agglomerates from different CeO_2-nanopowders with a primary particle size of approximately 20 – 30 nm. The number of smaller particles (particles below 300 nm) increases with increasing pressure difference, whereas the number of larger particles (particles above 300 nm) decreases due to deagglomeration. Additionally, scanning electron microscopy (SEM) is applied to study the morphology of the agglomerates with and without passing the orifice.

Experimental Setup



- generation of aerosol within pressurized beaker
- use of dry nitrogen → no moisture within carrier gas
- pre-separation of particles > ~1.5 µm within cyclone
- overpressure conditions: 20 kPa → 140 kPa in 20 kPa steps
- constant flow rate for all overpressure conditions (2 l/min)
- at 0 kPa overpressure: reference spectra

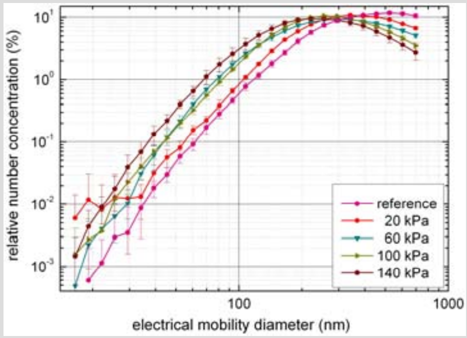
Data Evaluation



original data

(CeO_2, 63 g/cm³)

- for each pressure step: five SMPS-spectra averaged
- direct comparison of spectra difficult

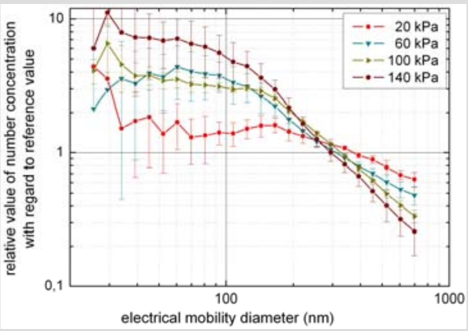


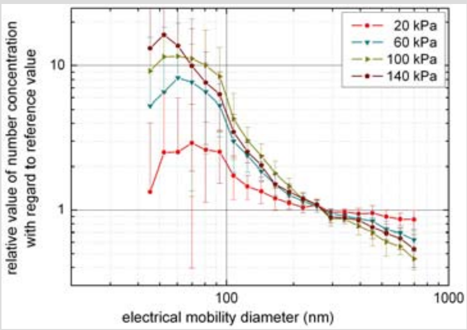
relative spectra

- calculation of relative spectra (normalized with respect to total concentration)
- using relative spectra: calculation of increase / decrease for a given size class (see results)

Results

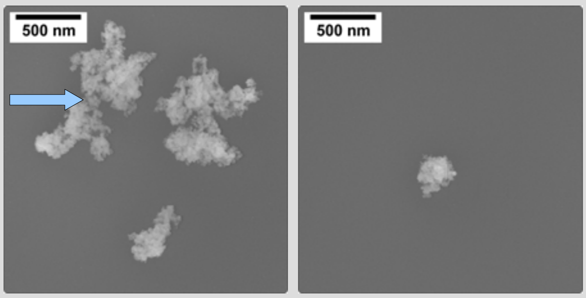
Example for deagglomeration of CeO_2 with a BET-surface of 63 m³/g (left) and 38 m³/g (right)





- error bars indicate worst case error (sum of standard deviations of reference spectra and „overpressure“ spectra)
- A clear shear force dependend shift towards smaller particles is observed
- for particles below approximately 300 nm: increase of particle number; for particles above approximately 300 nm: decrease of particle number
- dependent on particle properties (e.g. material, primary particle size, …)

Example for scanning electron micrographs of CeO_2-agglomerates before (left) and after (right) passing the orifice



- to investigate particle shape: deposition of agglomerates onto silicon substrates using electrostatic precipitation
- SEM images of agglomerates prior to deagglomeration, i.e. as prepared in pressurized beaker
- arrows indicate possible positions of break-up
- after passage of orifice usually more compact agglomerates

Conclusions

- Easy method to investigate stability of agglomerates consisting of primary particles with a diameter smaller than 100 nm
- With increasing overpressure increasing deagglomeration
- Relative increase or decrease of number concentration within certain size classes is obtained
- In combination with number concentration of reference spectra: method to investigate absolute values for the release of (nano-)particles under different stress levels
- Future tasks: Qualitative calculation of shear forces at different stress levels and calculation of bond energies for a given agglomerate size

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[1] B. Stahlmecke *et al.* (2008) EAC, Thessaloniki, Greece, T01A029O
[2] B. Stahlmecke et al., Investigation of nanopowder agglomerate stability in an orifice under various differential pressure conditions, submitted to JNR (2009)