Evaluation of Monodisperse Silver Particle Sintering Using a Tandem DMA Setup

Vinicius Berger, Hans-Joachim Schulz, Adam Boies, Eda Sorani, Jacob Swanson

Catalytic Instruments GmbH & Co. KG

27th ETH Nanoparticles Conference

Zürich, 14.06.2024

Silver Particles

Silver particles can be used as calibration aerosol for a variety of applications:

- Brake PN
- PTI
- RDE PEMS-PN

"thermally stable particles"

- ISO 27981: Calibration of CPCs
- ISO 15900: Determination of Particle Size Distribution
- CEN/TS 17434: Ambient air - Particle number size distribution of atmospheric aerosol
- CEN/TS 16976: Ambient air - Particle number concentration of atmospheric aerosol

"using spherical silver particles sized 40 nm produced by the evaporation/condensation method"

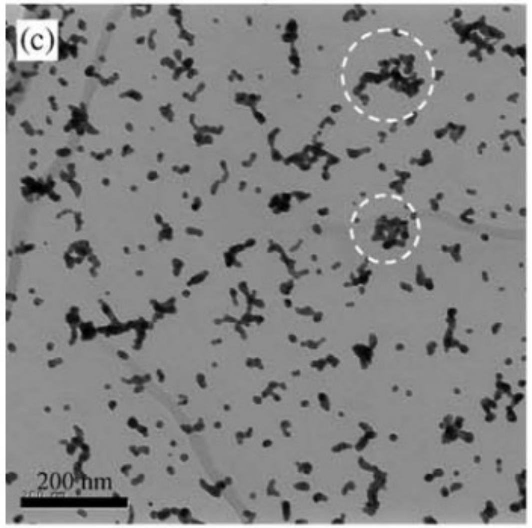
Silver Particle Sintering

Sintering:

- Heating of particles to change morphology
- Reshaping and compaction

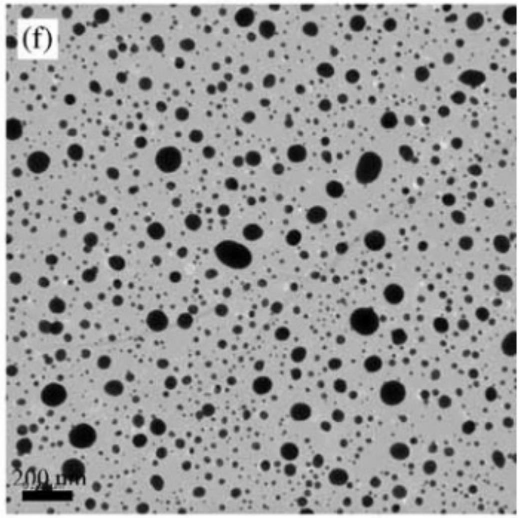
Goals:

- Achieving compact, thermally stable particles
  - Calibration of CPCs, PEMS, PTI
- Achieving spherical particles
  - Calibration of CEN-CPCs, DMAs



Ku et al., 2006

SINTERING



Ku et al., 2006

How to Generate Sintered Silver Particles

Tube furnace w. silver boat inside

= Scheibel-Porstendörfer method

&

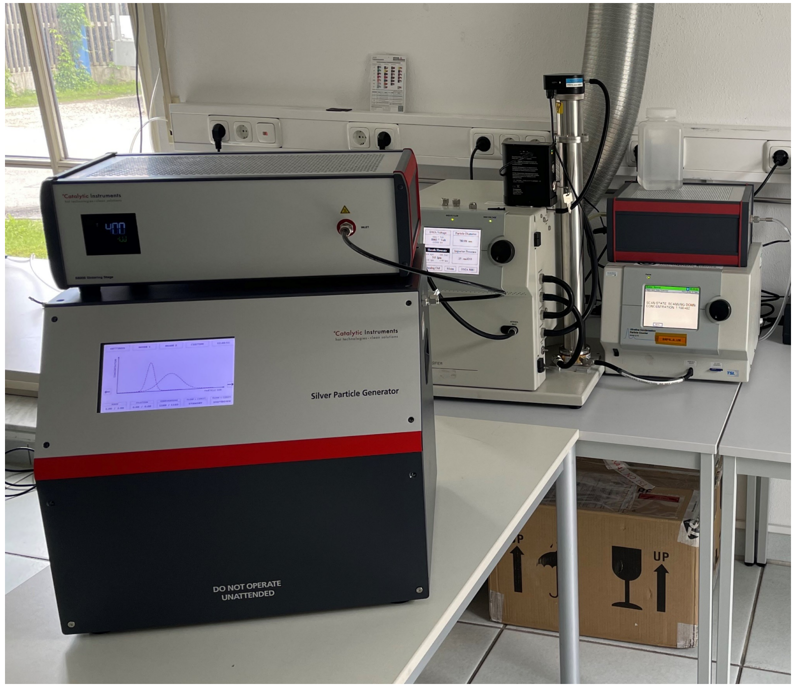
2^{nd} tube furnace to sinter particles

Silver Particle Generator (SPG)

&

Particle Sintering Device S8000

Which sintering temperature and residence time is necessary?



How to Generate Sintered Silver Particles - Literature

Generation and investigation of airborne silver nanoparticles with specific size and morphology by homogeneous nucleation, coagulation and sintering

Bon Ki Ku*, Andrew D. Maynard

Ku et al. 2006

- First sintering effects start at 100 °C
- No change in electrical mobility for 500 °C, 700 °C, 800 °C

Seeded growth of monodisperse and spherical silver nanoparticles

CrossMark

Simon Zihlmann*, Felix Lüond, Johanna K. Spiegel

Zihlmann et al., 2014

- T_{sinter} = 500 °C (Gold particles)

AEROSOL SCIENCE AND TECHNOLOGY
2016, VOL. 50, NO. 4, 331–338
http://dx.doi.org/10.1080/02786826.2016.1152351
Taylor & Francis
Taylor & Francis Group

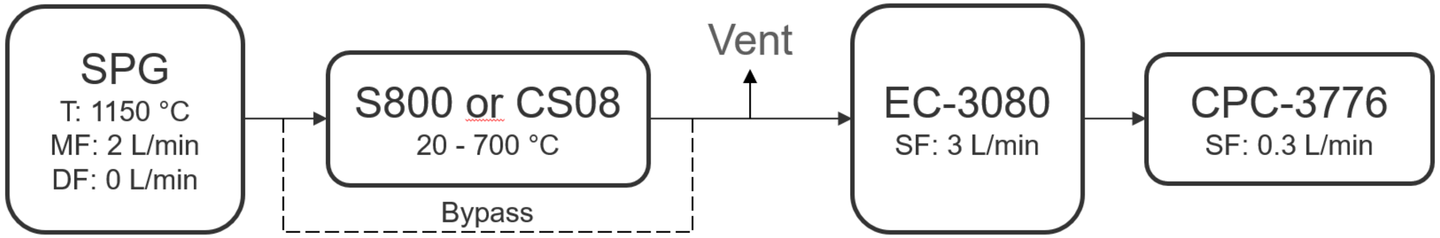
Dependence of CPC cut-off diameter on particle morphology and other factors

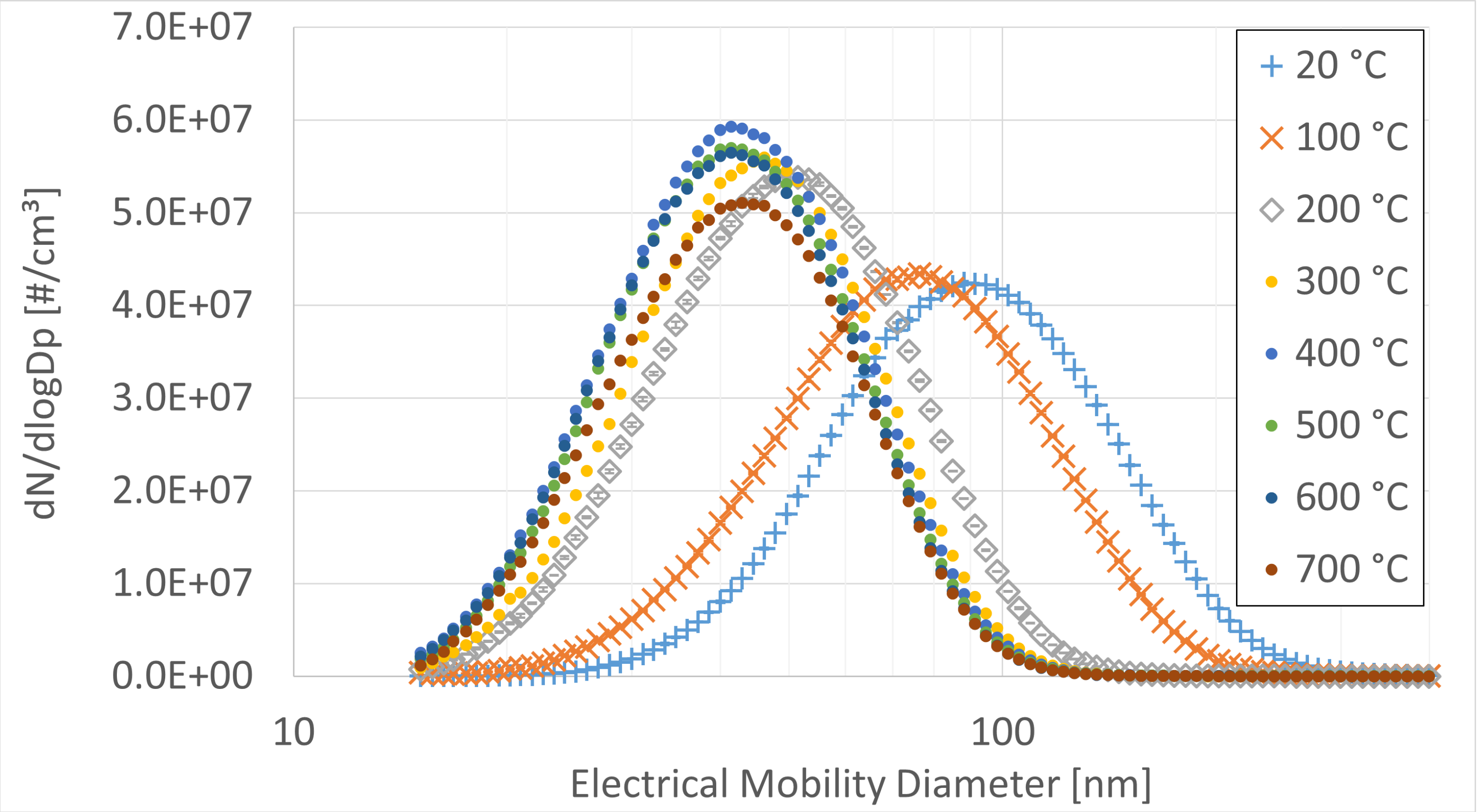
Thomas Tuch[a], Kay Weinhold[a], Maik Merkel[a], Andreas Nowak[b], Tobias Klein[b], Paul Quincey[c], Mark Stolzenburg[d], and Alfred Wiedensohler[a]

Tuch et al., 2016

- T_{sinter} = 450 °C
- Secondary sintering at 350 °C

Sintering Investigation – SPG & S800 / CS08





Asfera CFA Paris 2024

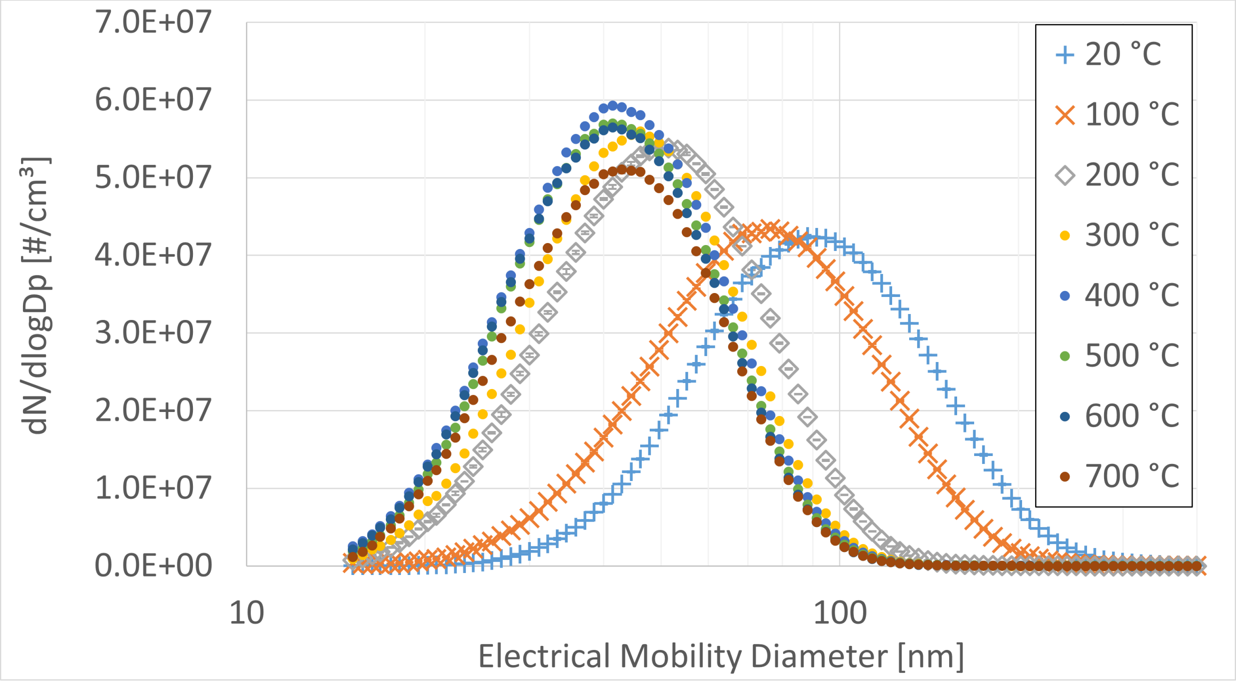
- First sintering effects at 100 °C
- No change in GMD for 400, 500, 600, 700 °C

➪ Confirming results from Ku et al., 2006

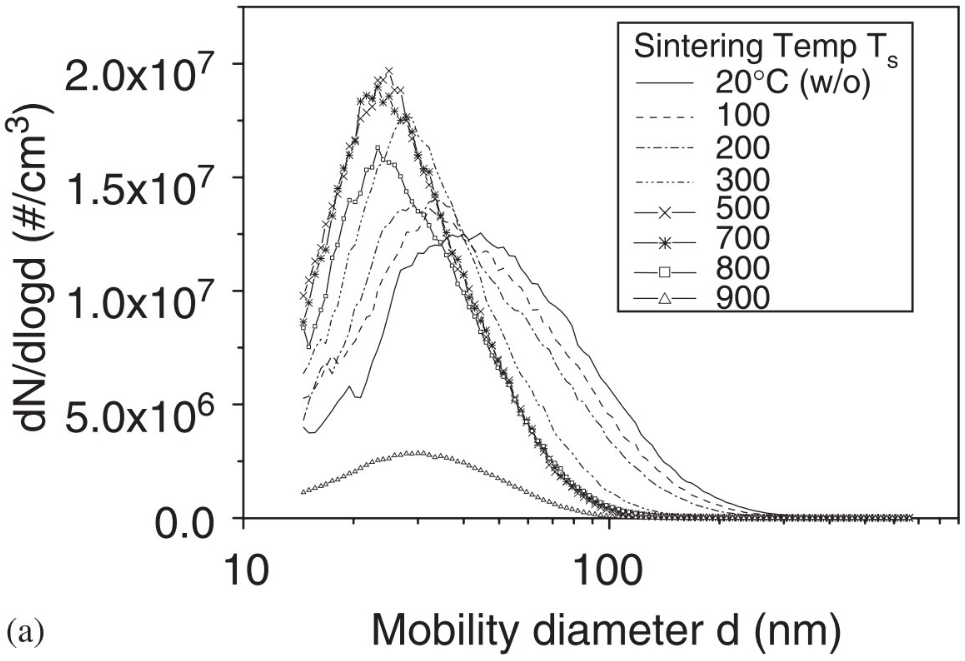
➪ S800 & CS08 work as sintering devices!

➪ "Spherical silver particles sized 40 nm" achievable!

Sintering Investigation – SPG & S800 / CS08



Ku et al., 2006



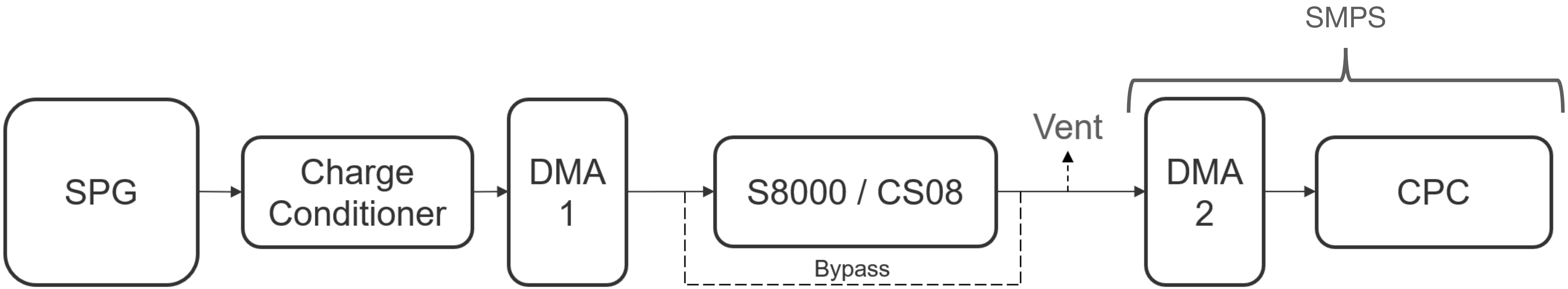
(a)

- First sintering effects at 100 °C
- No change in GMD for 400, 500, 600, 700 °C

➪ confirming results from Ku et al., 2006

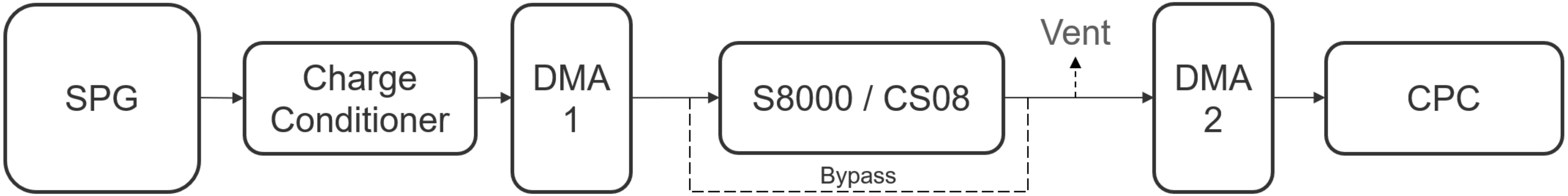
More detailed analysis wanted!

Sintering Investigation – Tandem DMA Setup

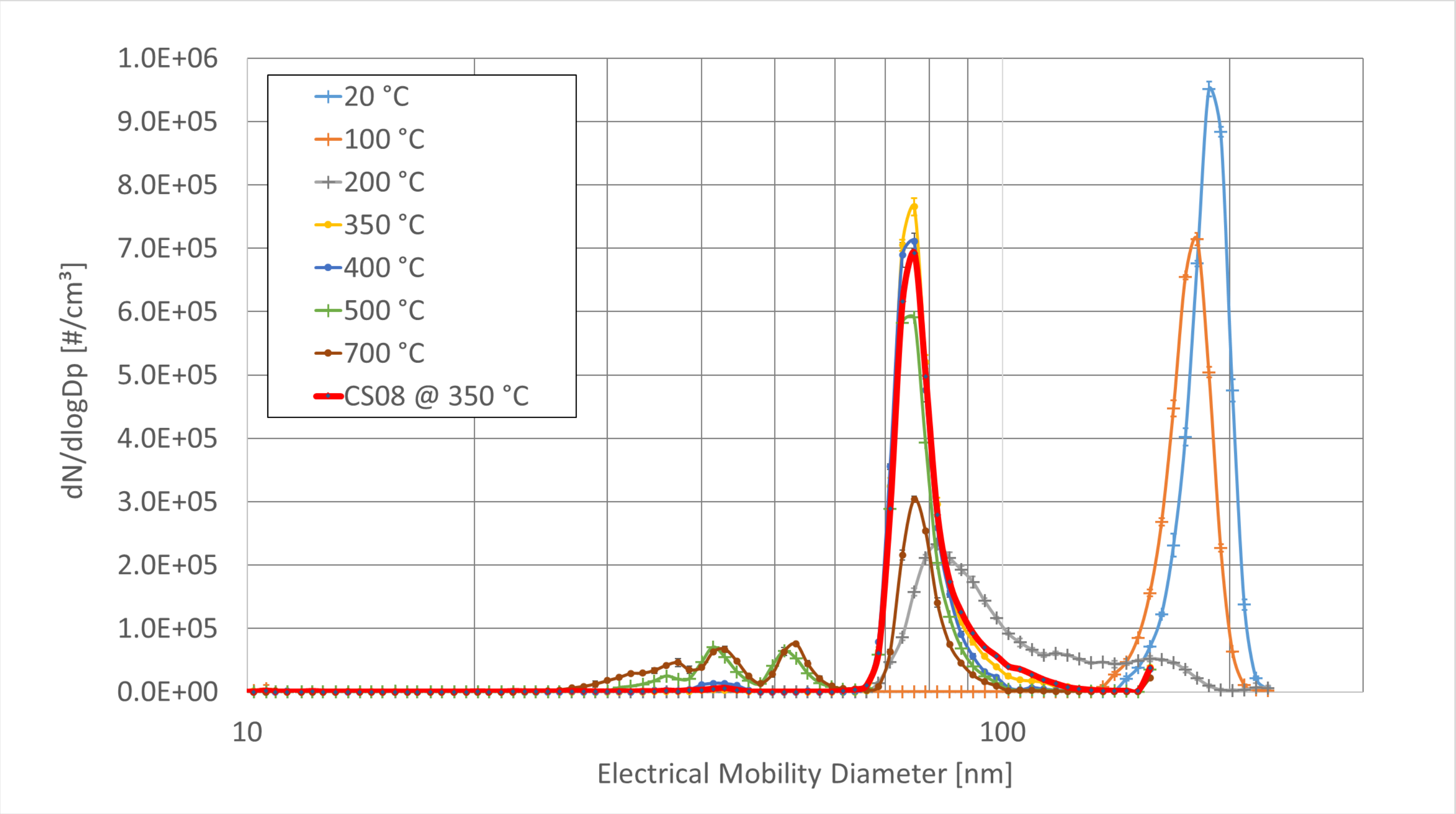


SPG	Charge Conditioner	DMA 1	S8000 / CS08	DMA 2	CPC
CI Silver Particle Generator **T = 1150 °C** Flow = 1.55 L/min (Air)	TSI 3088 X-Ray Neutralizer	TSI 3080 Electrostatic Classifier TSI 3081 LDMA Sheath Flow = 15.5 L/min **d_{set} = 200 nm**	Particle Sintering Device S8000 T = 20 – 700 °C **t_{res} = 9 – 2.6 s** / Catalytic Stripper CS08 T = 350 °C **t_{res} = 1.5 s**	TSI 3080 Electrostatic Classifier TSI 3081 LDMA / TSI 3085 NDMA Sheath Flow = 15 L/min	TSI 3776 UCPC d_{50} = 4 nm Flow = 1.5 L/min

Sintering Investigation – Tandem DMA Setup



Sintering Investigation – Tandem DMA Results

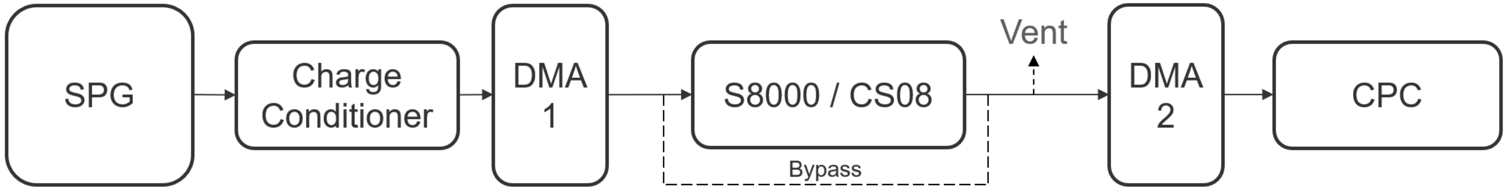


- First sintering effects at 100 °C
- No change in GMD for 400, 500, 600, 700 °C

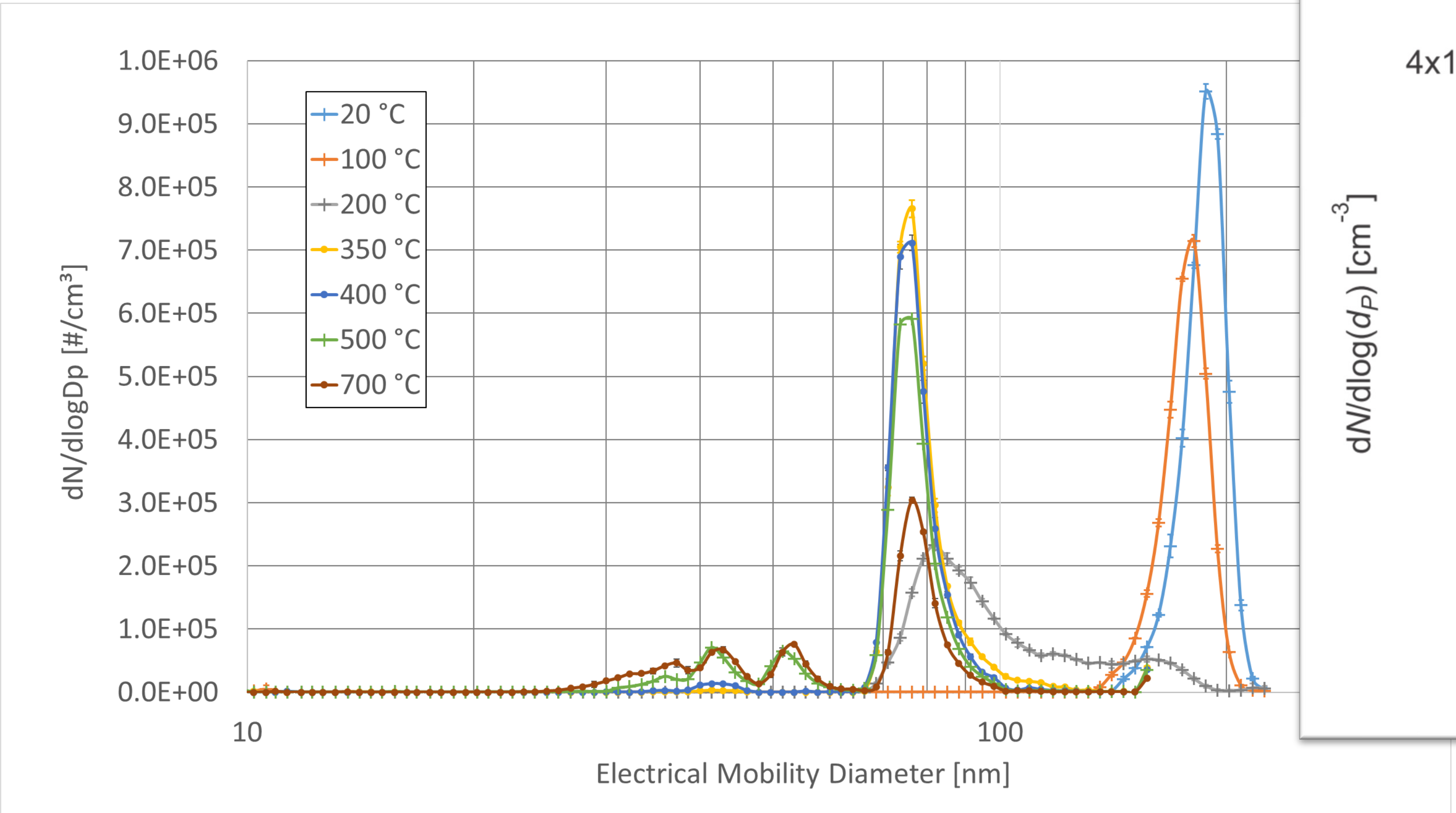
⇨ confirming results from Ku et al., 2006

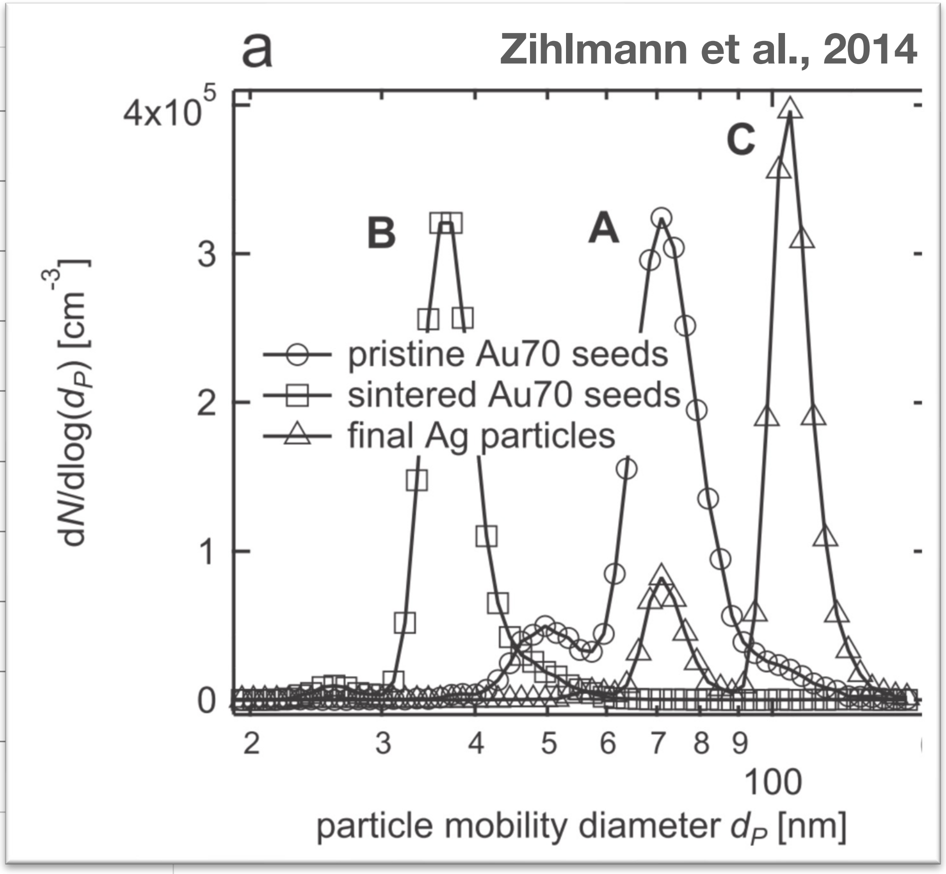
⇨ **S8000** & CS08 work as sintering devices!

⇨ spherical silver particles sized 40 nm achievable!



Sintering Investigation – Tandem DMA Results





- Reduction in electrical mobility diameter ≈ 60 %, in good agreement with Zihlmann et al., 2014
- 200 nm particles collapse into 75 nm particles (spheres? TEM images needed!)

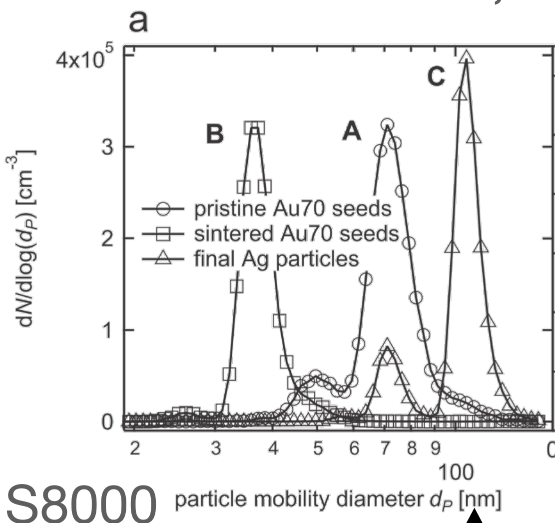
Silver Particle Sintering – Summary

- ✓ Combination of SPG and S8000 generates sintered, thermally stable silver particles with up to 75 nm electrical mobility diameter
- ✓ Resulting aerosols are similar to a classical double tube furnace setup
- ✓ Sintering behavior described by Ku et al., 2006, confirmed:
  - First sintering occurs around 100 °C
  - Above 400 °C sintering temperature no change in electrical mobility diameter observable

  ⇨ CI recommends 400 °C as S8000 sintering temperature

Zihlmann et al., 2014

Silver Particle Sintering – Outlook



- Confirm particle size and morphology with TEM images
- Investigate influence of sintering temperature on 23 nm-CPC d_{50}*
- Investigate how to generate larger sintered silver particles with SPG & S8000
  - Higher generator temperatures up to 1500 °C are currently investigated**
  - Heterogeneous condensation of silver (Tandem SPG)
- Reduce agglomeration after sintering
  - ➪ dilute directly after sintering i. e. inside S8000 (+ reduces thermophoretic losses)
- Investigate high-effort & high-quality calibration approach:
  - Generator → Size selection → Sintering → CPC & FCAE (& SMPS for size confirmation)

* = Tuch et al., 2016
** = Please feel free to suggest other materials!

Contact:
vinicius.berger@catalytic-instruments.com