

# Freeze Granulation for Processing of Nano Materials

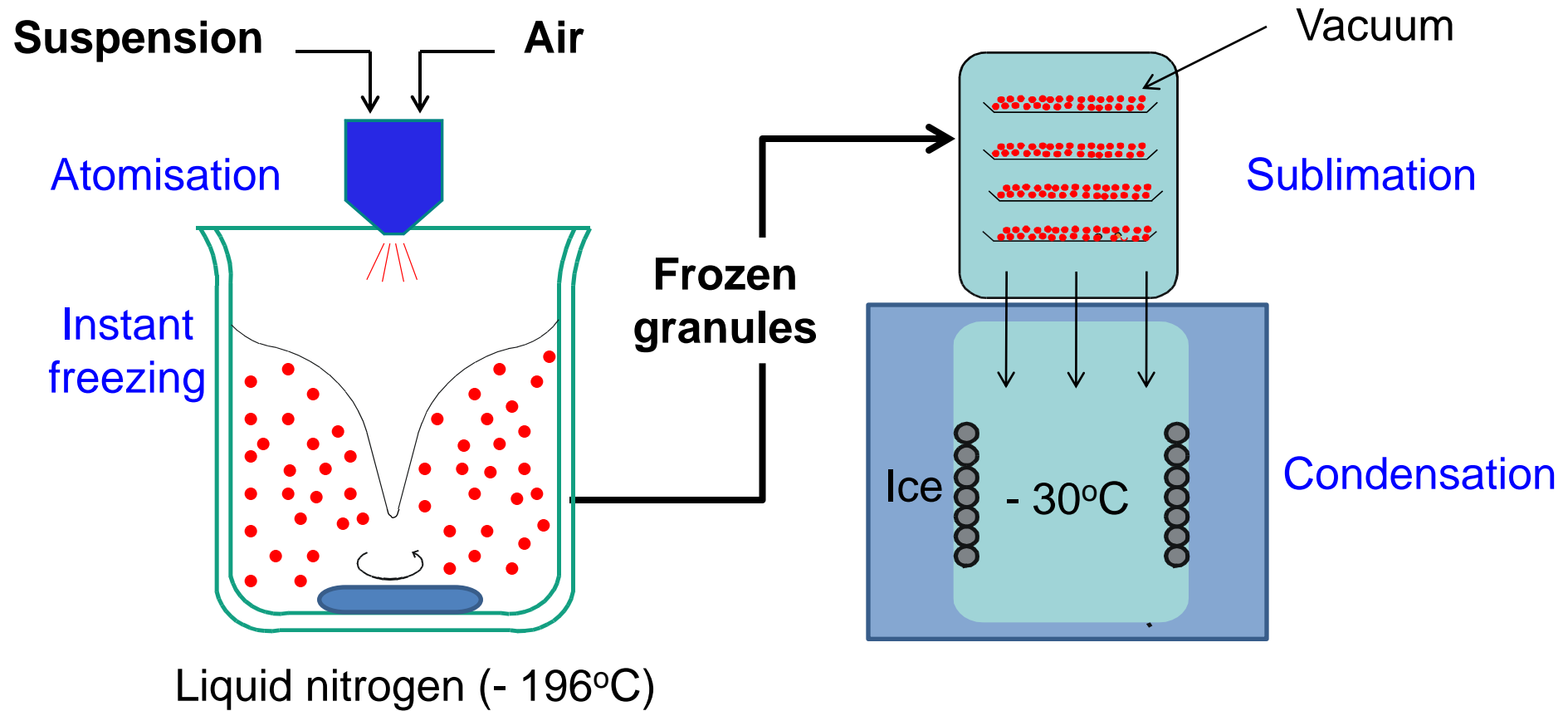
Kent Rundgren, President of PowderPro AB

Sweden

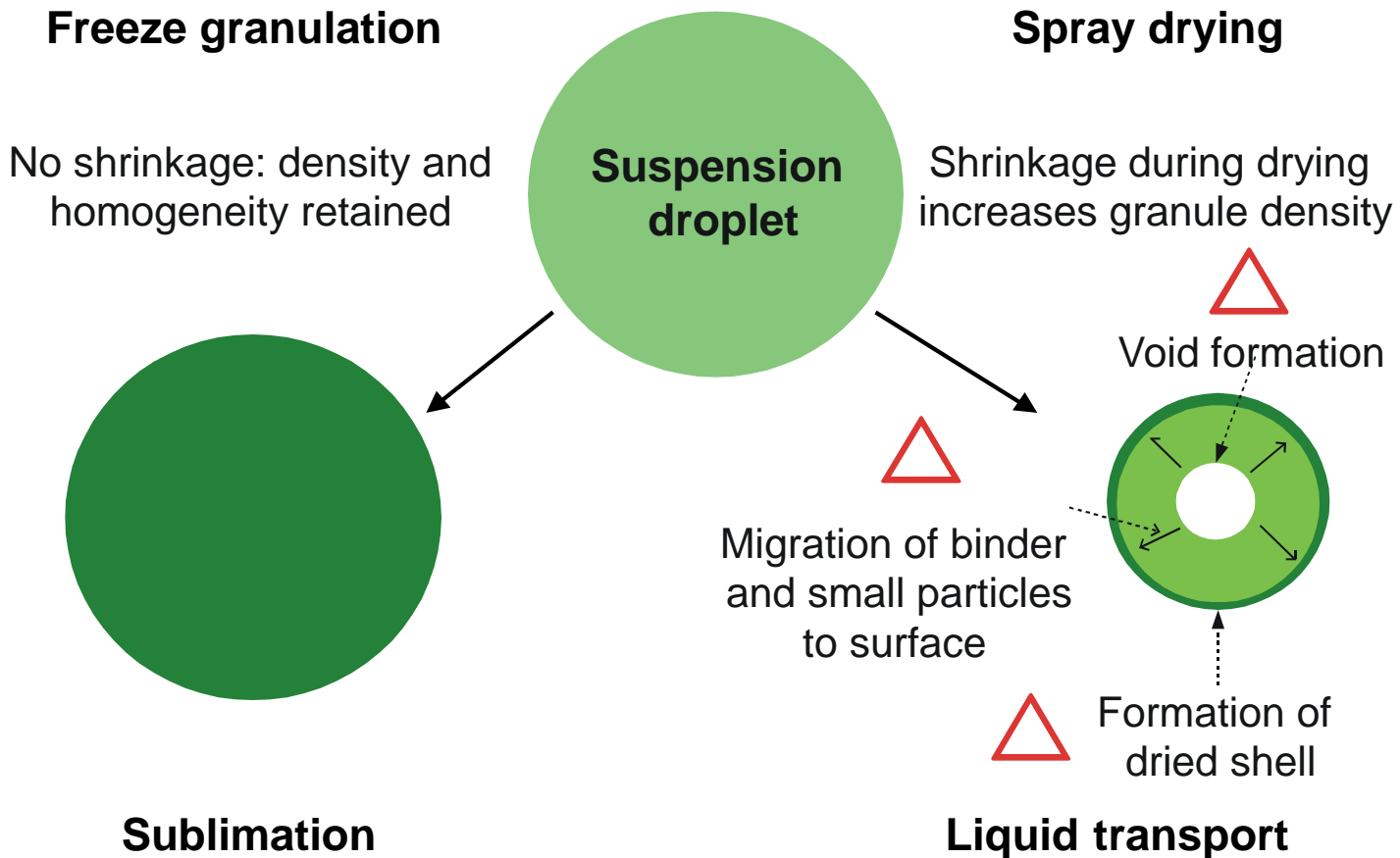


# Freeze Granulation Process

**Spray freezing** → **Freeze drying**



# Freeze Granulation vs Spray Drying

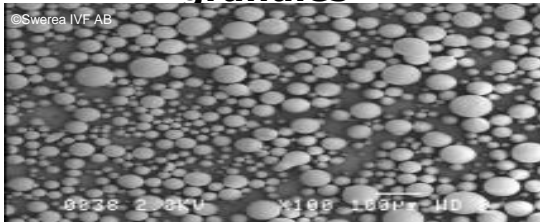


# Freeze Granulation

A variety of applications

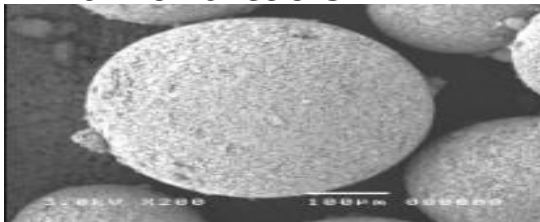
**Atomisation**

**Homogenous granules**



Metal powders  
Explosives

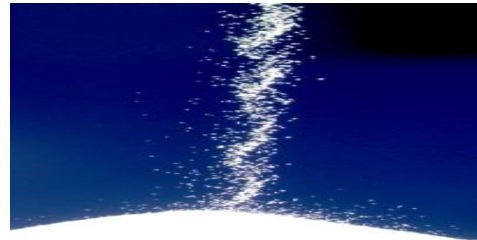
Diamond tools



*Composites*

Chemical Looping  
Combustion

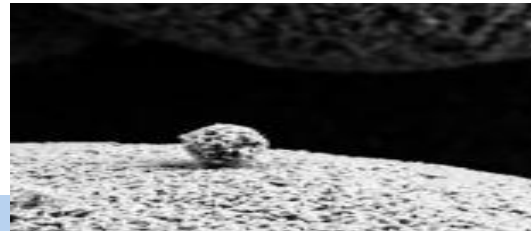
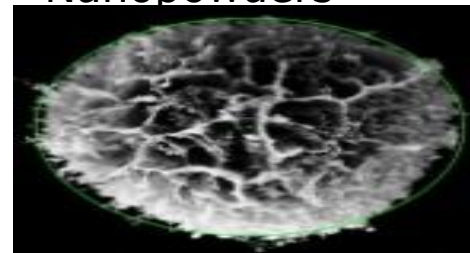
*Material synthesis*



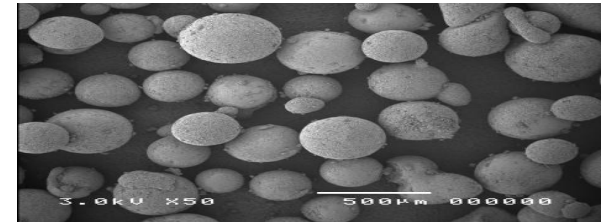
**Bioceramics**

LED-Lighting materials

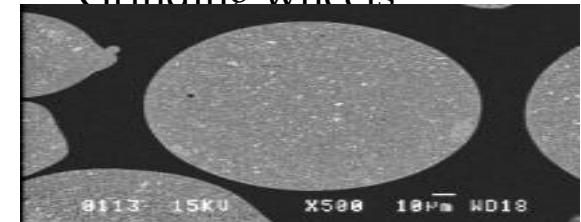
Nanopowders



Instant freezing  
Spherical  
Free flowing



Ceramics  
*Biomaterials*  
Grinding wheels



*Catalysts*

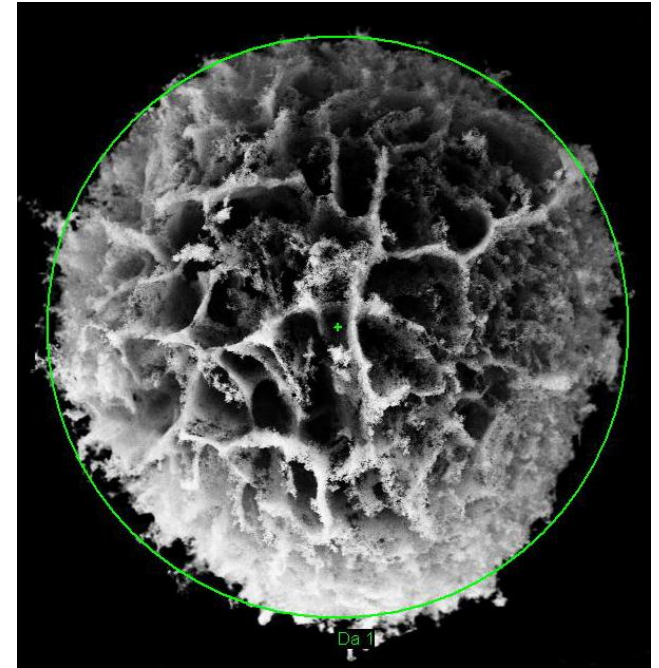
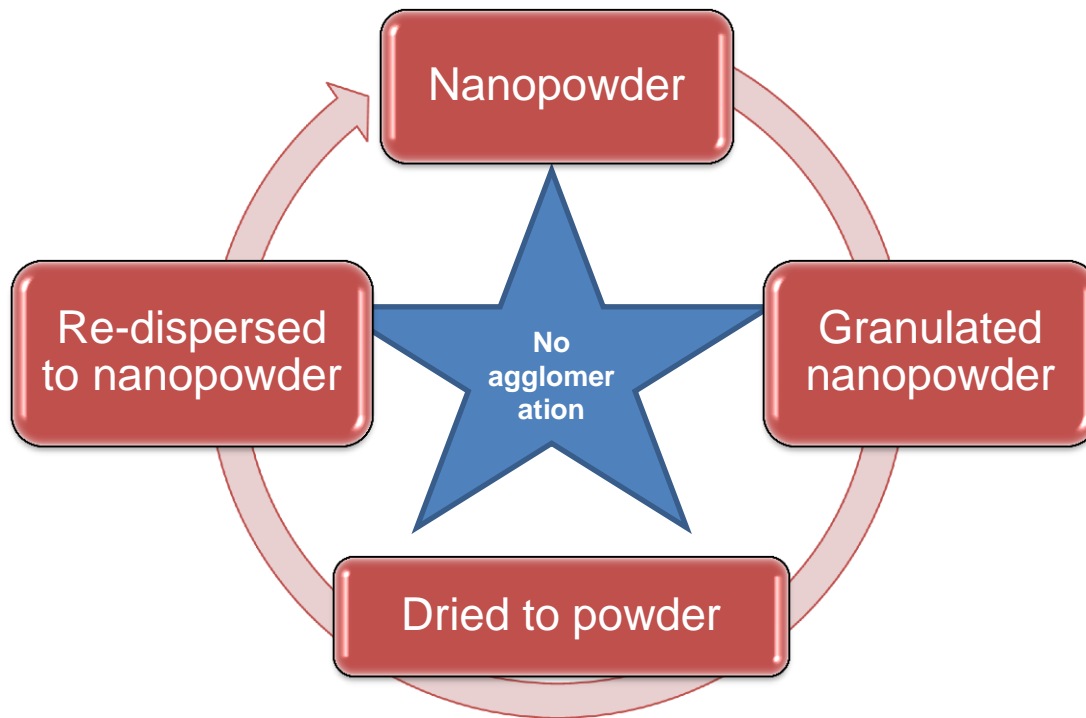
**Nanomaterials**

Pharmaceuticals

Chemicals

**Freeze Granulation – The Way to Optimised Powder Processing!**

# Freeze Granulation of nanopowders



- Unique process to retain nanomaterial particle size
- Nano-particles keep separated
- Re-dispersible end-result

# Freeze Granulation

- Method to preserve nano properties
- Method to produce *homogeneous* granules and preserve material homogeneity
- Method to avoid strong bonding and oxidation
- Method to produce granules of nano materials that can be handled and processed in dry state
- Method to produce granules, easy to re-disperse to original particle size distribution

# PowderPro AB

Founded in 2000, as a spin-off company from the Swedish Ceramic Institute, to provide equipment for freeze granulation

- a privately owned, expanding company
- science-based
- affiliated to Swerea IVF AB and Chalmers Industriteknik
- especially experienced in the ceramic and powder metallic fields
- looking to expand into fields where high-quality freeze granulation can improve processes and output quality

# Freeze Granulator



Models: LS-2  
and LS-6

Small scale with capacity to process up to 6 litre suspension per hour

Medium scale with capacity of processing 25 litre per hour is under development

# Freeze drier GT-2



Heated trays for quick drying

Capacity to dry ca 2 litre of granules that is achieved from ca 1 litre powder suspension

Driers with much higher capacity exist on the market

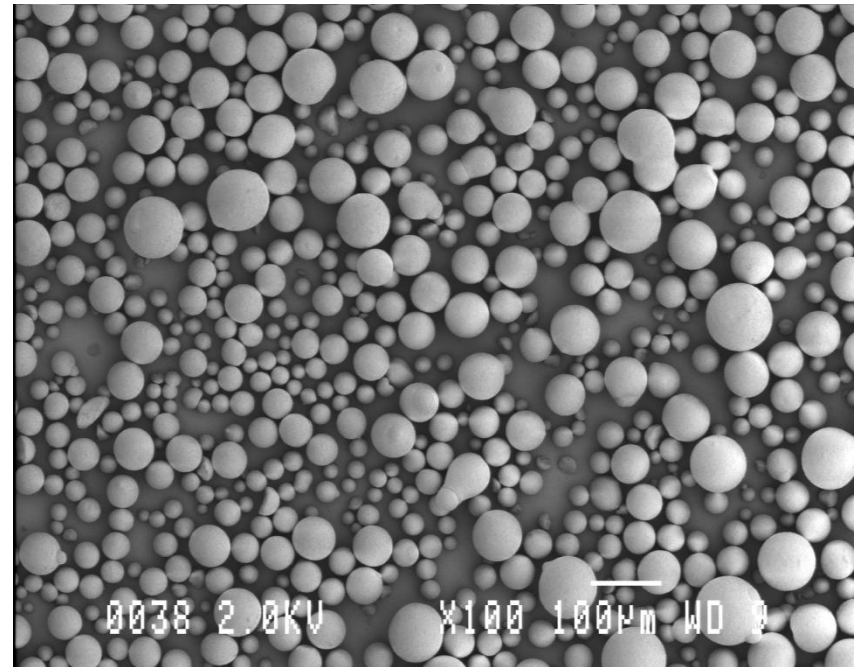
# Aspects in processing of small units of matter

- Difficulties to retain homogeneity during processing
- Difficulties to keep small units separated
- Drying from suspension causes agglomeration, segregation and oxidation
- Difficulties to obtain handable/processable nanopowders in dry state
- Difficulties to re-disperse from dry state

# Freeze Granulation Process Benefits

- Homogeneous granules
  - *No cavity or void formation*
  - *No migration of any substances or small particles to surface*
  - *No, or limited, shrinkage of granules*
  - *No formation of a dried shell*
- Granule density controlled by suspension composition
- Vacuum drying minimizes oxidation
- High yield, low waste of material
- Process allows latex (water insoluble polymers) as binder
- Recycling of solvent possible

# Granule properties



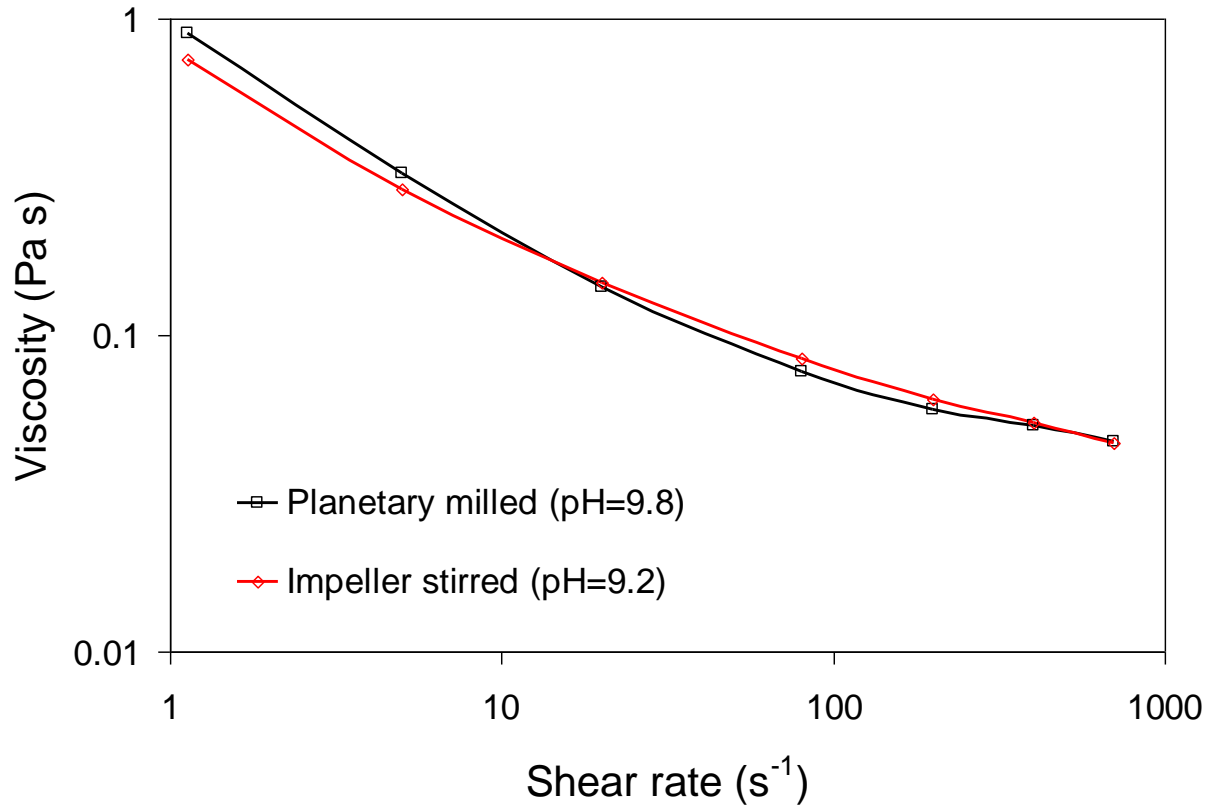
Free-flowing granules with a wide granule size distribution (20–500  $\mu\text{m}$ )

Density depends on solids in suspension

Size depends on suspension viscosity and granulation parameters

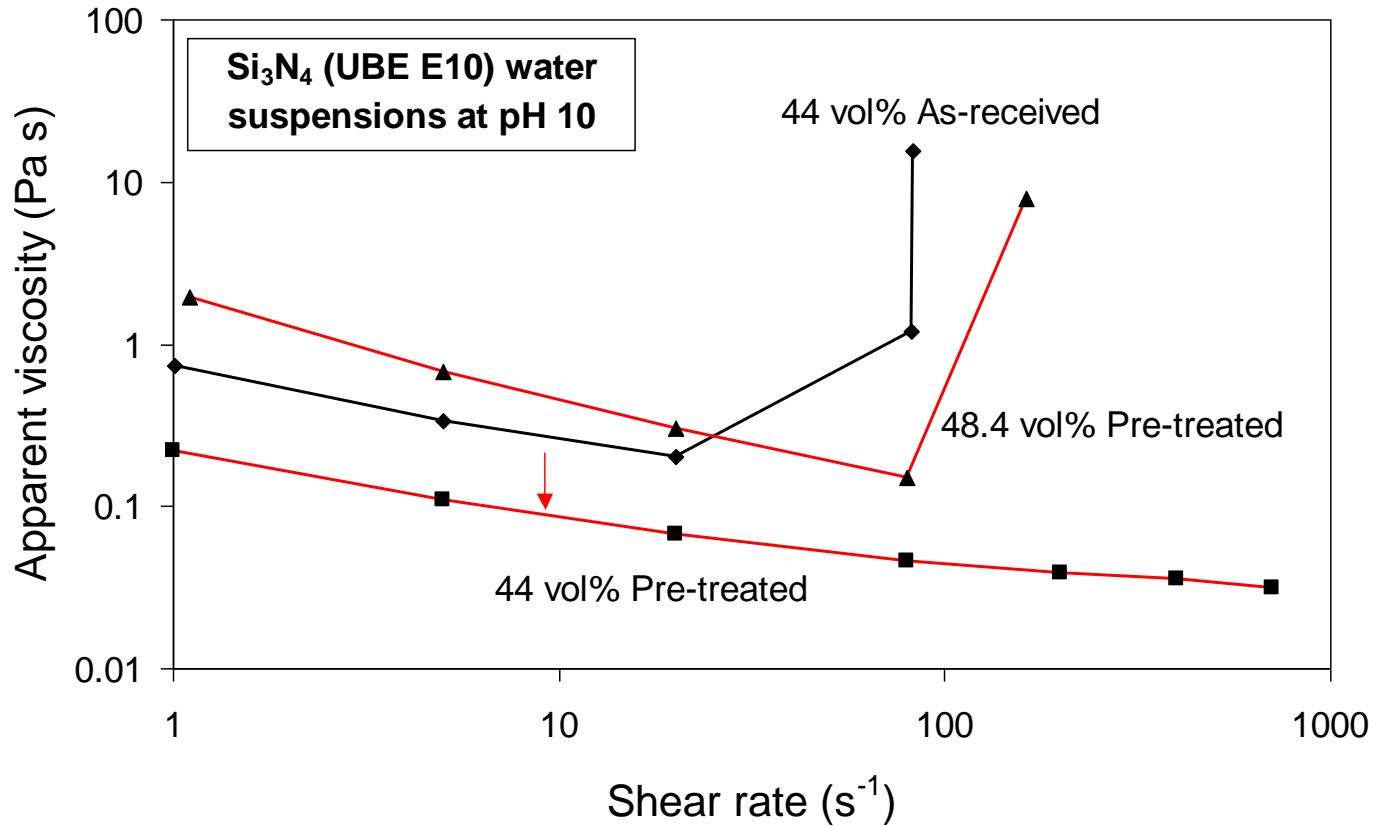
# Re-dispersibility of submicron powders - 1

Freeze granulated/freeze-dried powders can be re-dispersed with impeller stirring to the same de-agglomerated state as with planetary ball milling

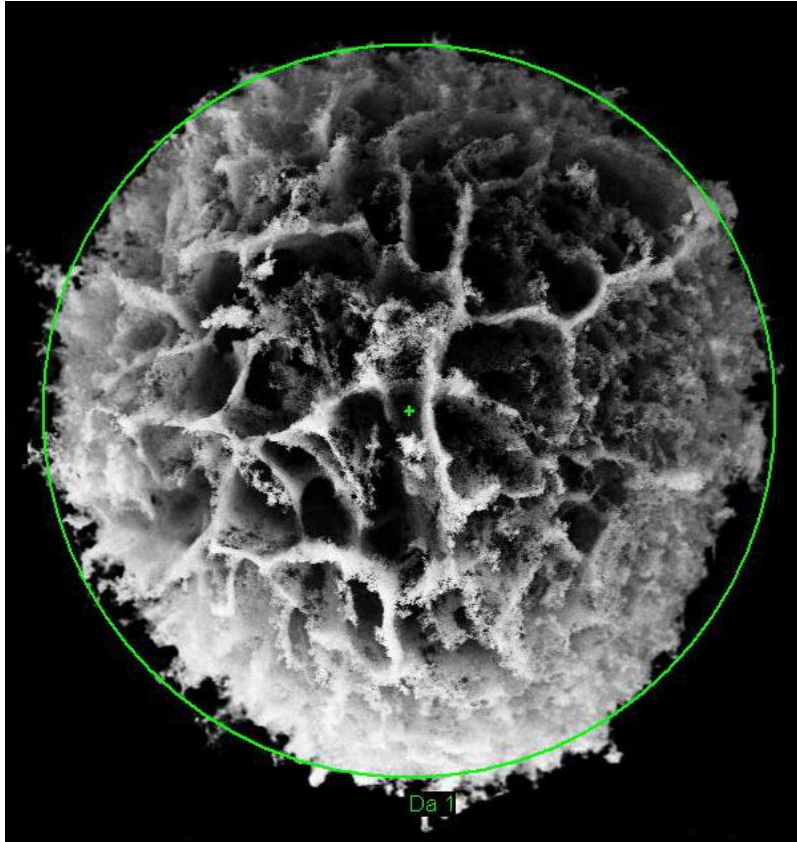


# Re-dispersibility of submicron powders - 2

Powders, difficult to deagglomerate, can be dispersed at low concentrations, freeze granulated/freeze-dried and re-dispersed at higher concentrations



# Granules of "nano"powders

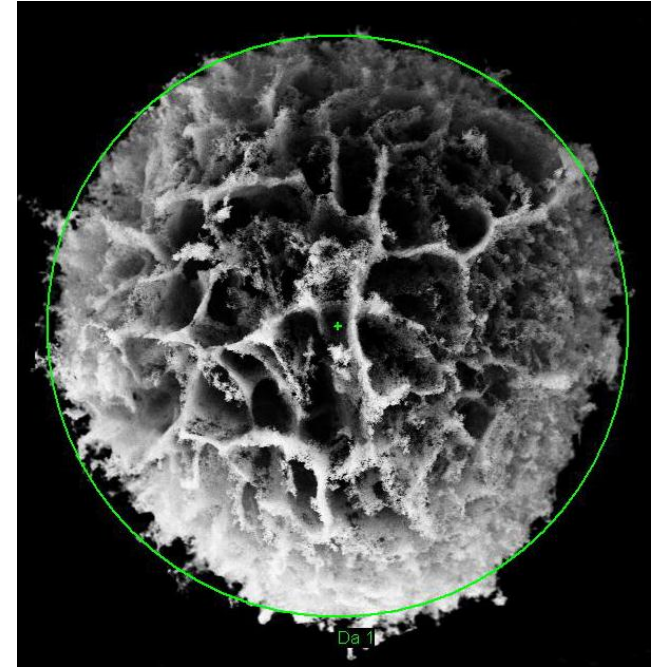
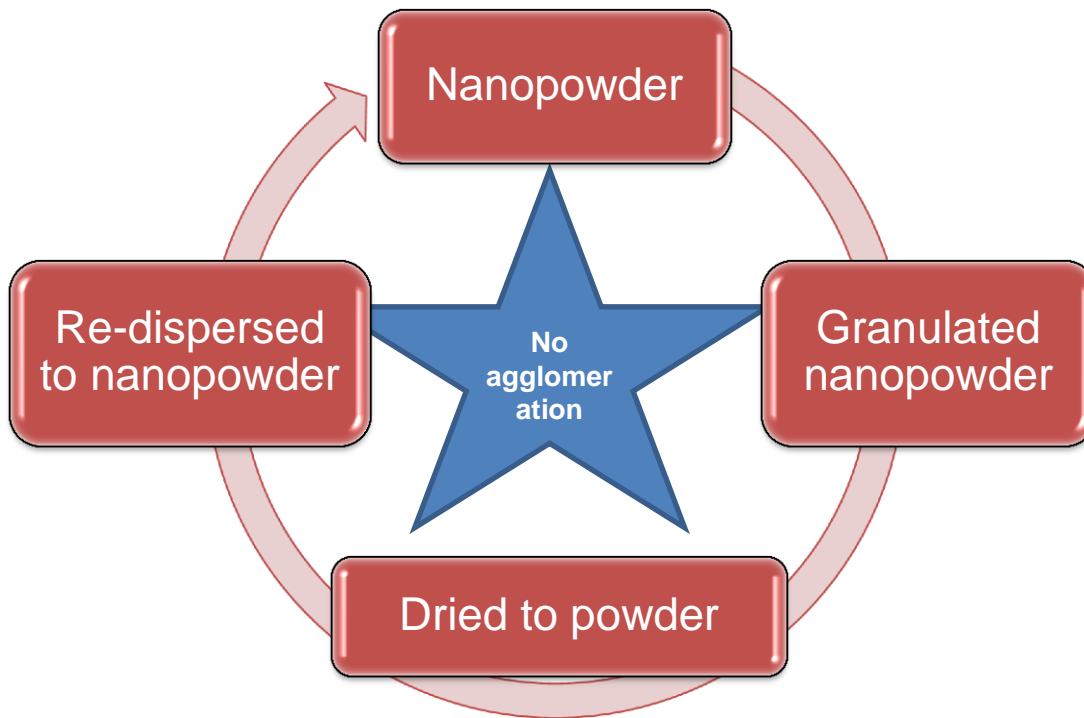


Granule based on 10-30 nm particles at low concentration



Granule based on 500 nm particles at high concentration

# Freeze Granulation of nanopowders



- Unique process to retain nanomaterial particle size
- Nano-particles keep separated
- Re-dispersible end-result

# Various aspects and uses

- All kinds of material in dispersed form can be granulated
- Ideal for preparation of fine powder mixes with suitable additives for subsequent processing
- Suspension quality always determines and will reflect the granule quality in terms of homogeneity
- Favorable preparation step for material synthesis owing to the homogeneity of the granulated material
- Water as medium is preferred but also organic solvents with suitable freezing point (-25 to +10°C) can be used

# PowderPro: Offers and Services

- Supplies freeze granulation equipments for Lab, Pilote-scale and production
- Carry out test granulations of customer-supplied materials
- Supports process implementation and start-up with customers

PowderPro has close collaboration with Swerea IVF AB in research related to freeze granulation in a wide range of material/applications

# Mission

**PowderPro's mission is to provide first-class Freeze Granulation and drying techniques for processing of ceramic and metal powders, nanomaterials, diamonds, proteins, enzymes, fine chemicals and pharmaceuticals.**

# Freeze Granulation - Summary

- provides a competitive technique for the manufacture of granules for various purposes
- preserves the material homogeneity
- enhances subsequent processing performance
- enables equally appropriate process for smaller and larger quantities with equal results
- enables controlled granule density can be
- provides mild drying with low degree of oxidation