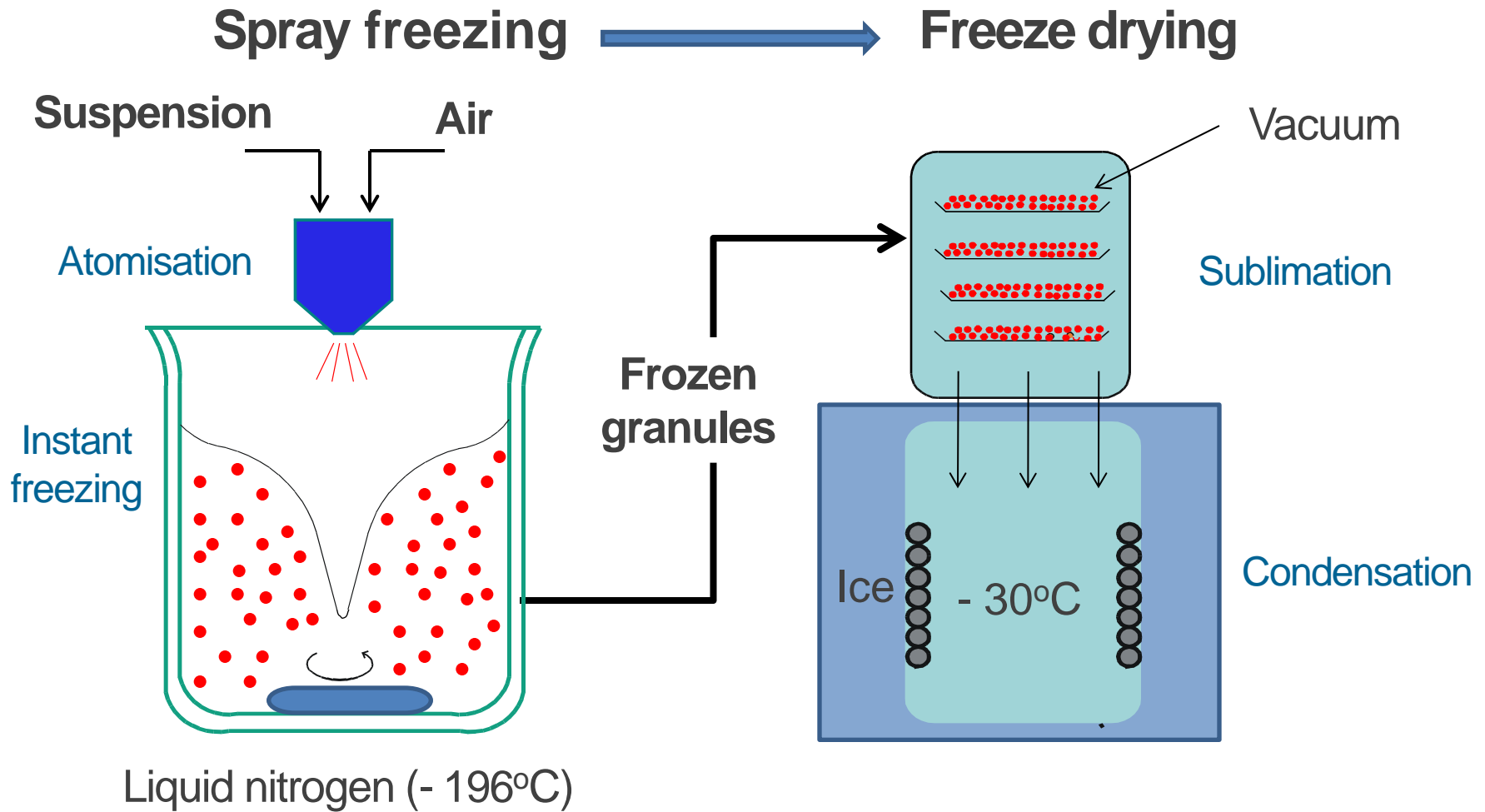




# Freeze Granulation for Processing of Nano Materials

**Kent Rundgren, President of PowderPro AB**

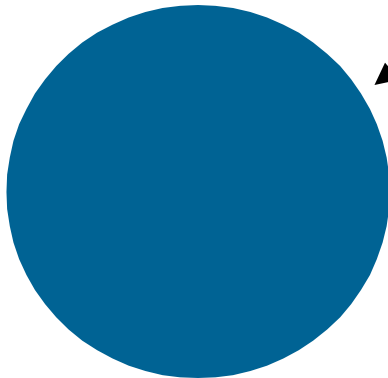
# Freeze Granulation Process



# Freeze Granulation vs Spray Drying

## Freeze granulation

No shrinkage: density and homogeneity retained

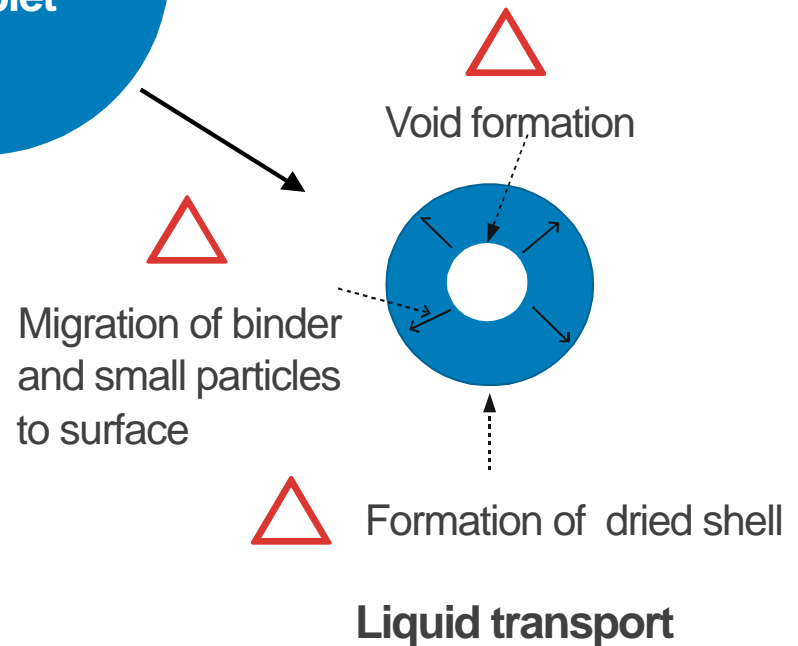


**Sublimation**

## Suspension droplet

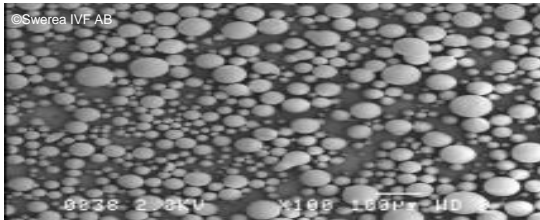
## Spray drying

Shrinkage during drying increases granule density

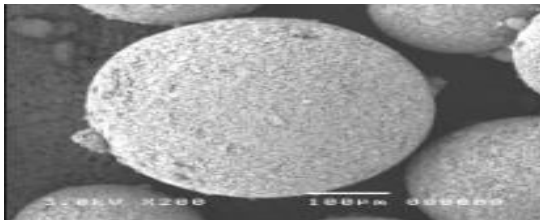


# A variety of applications

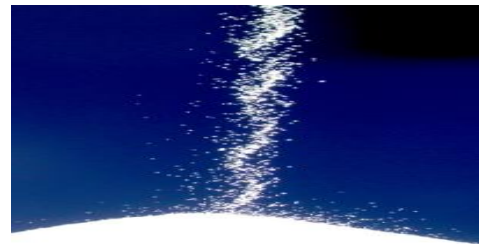
**Atomisation**  
**Homogenous granules**



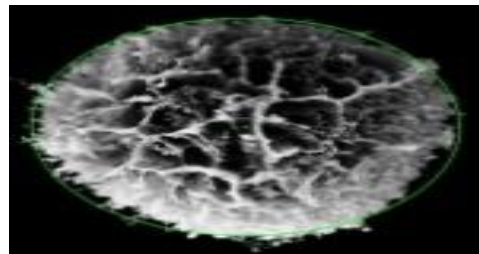
**Metal powders**  
**Explosives**  
**Diamond tools**



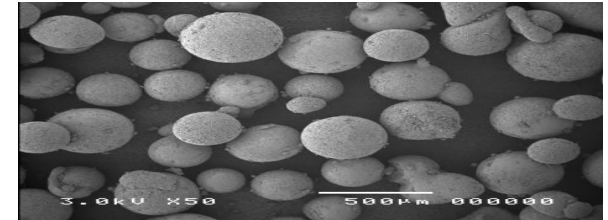
**Composites**  
**Chemical Looping**  
**Combustion**  
**Material synthesis**



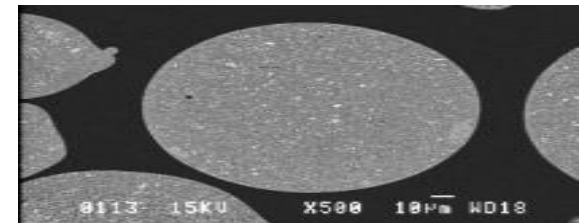
**LED-Lighting materials**  
**Nanopowders**



**Instant freezing**  
**Spherical Free flowing**

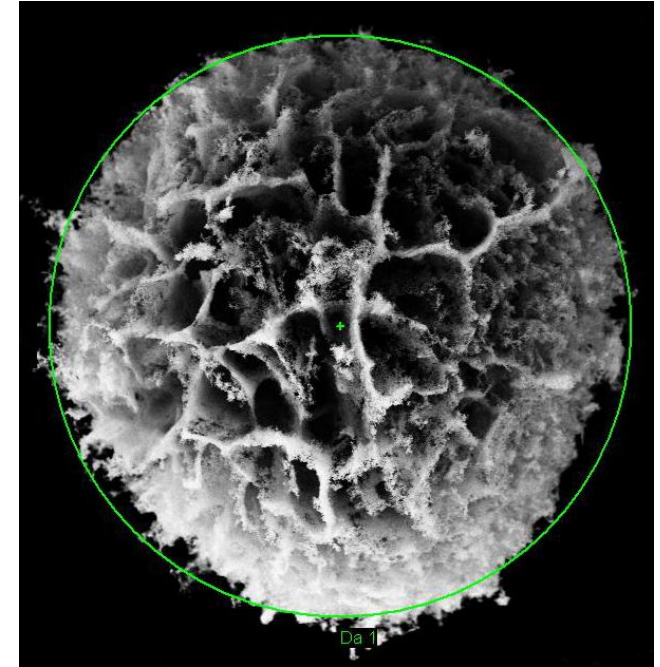
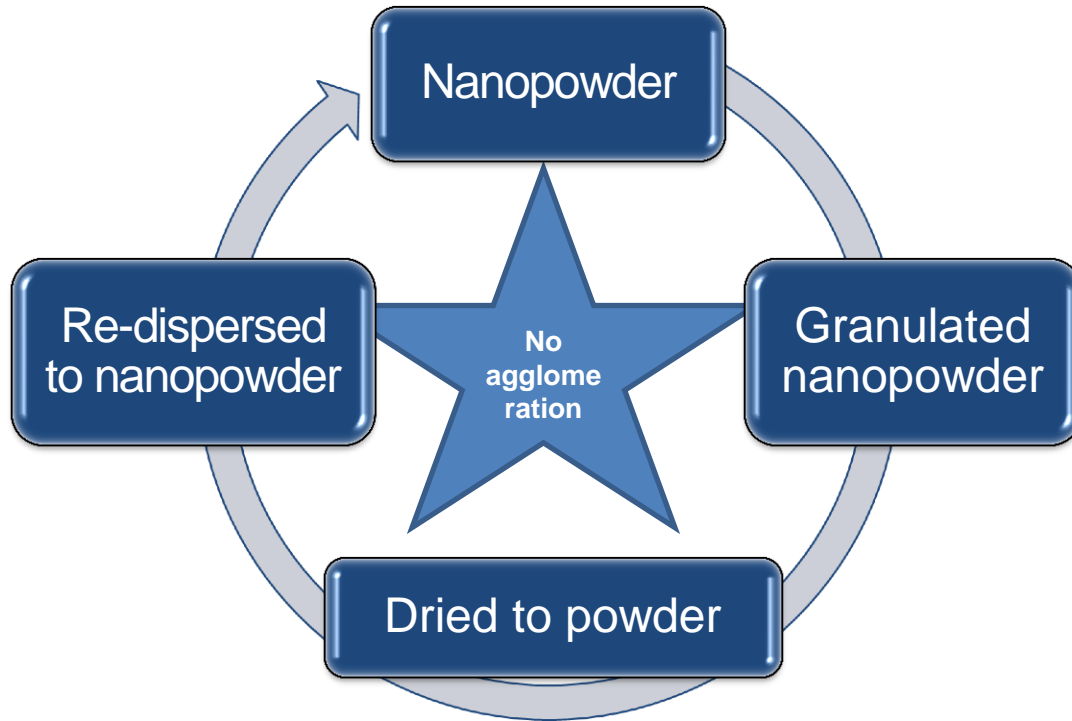


**Ceramics**  
**Biomaterials**  
**Grinding wheels**



**Catalysts**  
**Nanomaterials**  
**Pharmaceuticals**  
**Chemicals**

# 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 Granulation

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

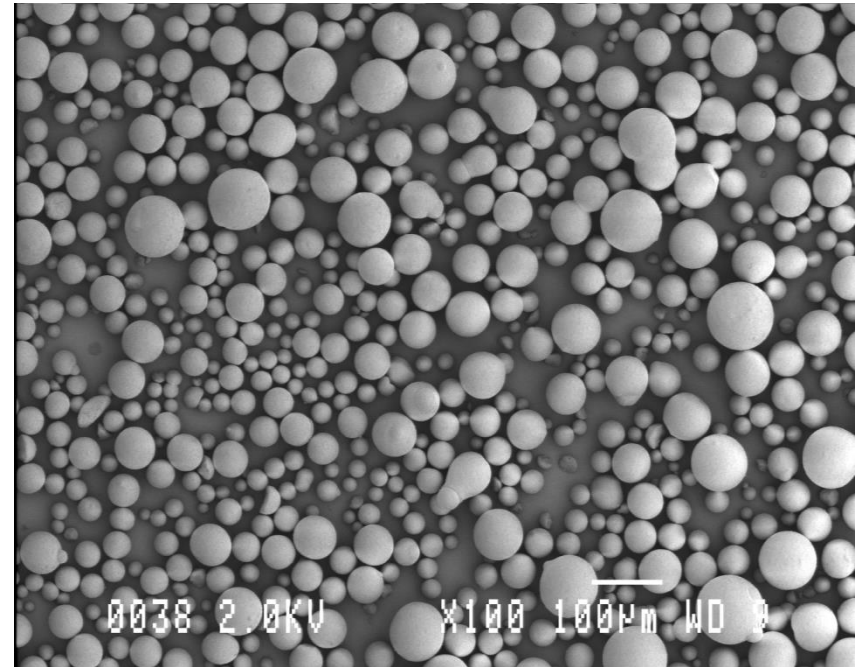
# Aspects in processing of small units of matter

- » 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



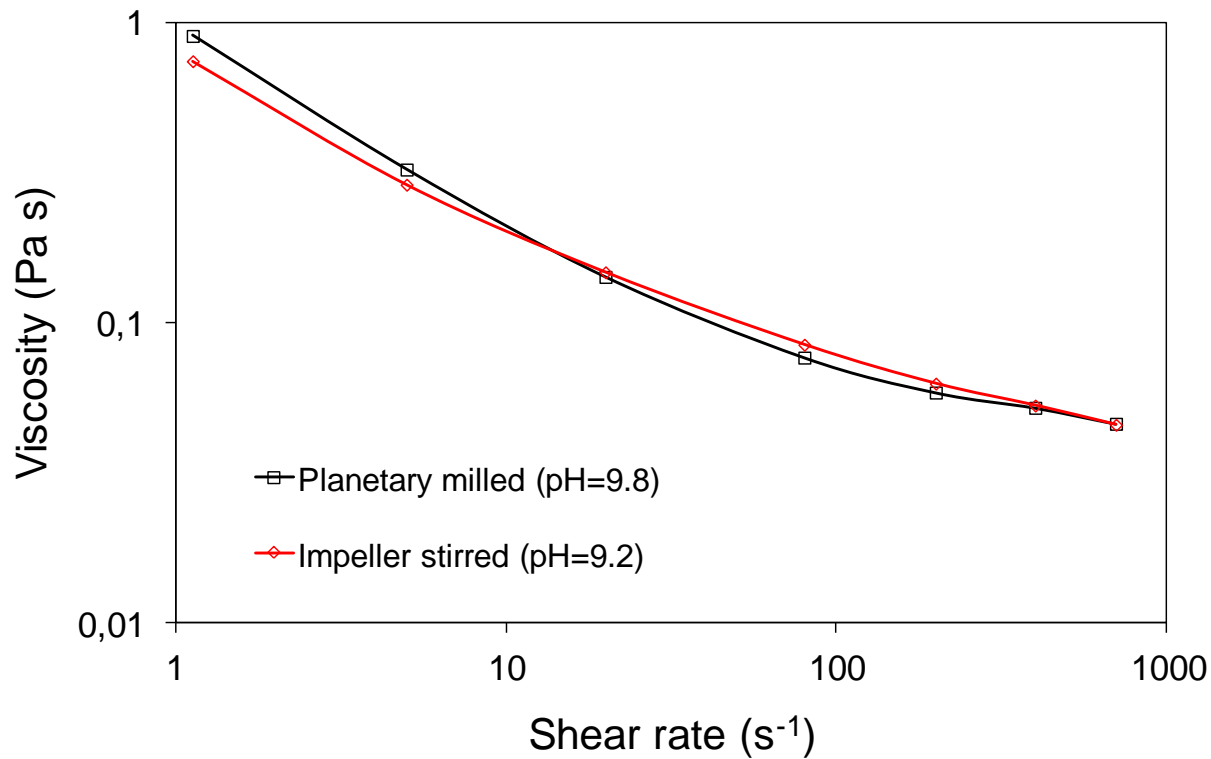
Free-flowing granules with a wide granule size distribution (20–500 μm)

Density depends on solids in suspension

Size depends on suspension viscosity and granulation parameters

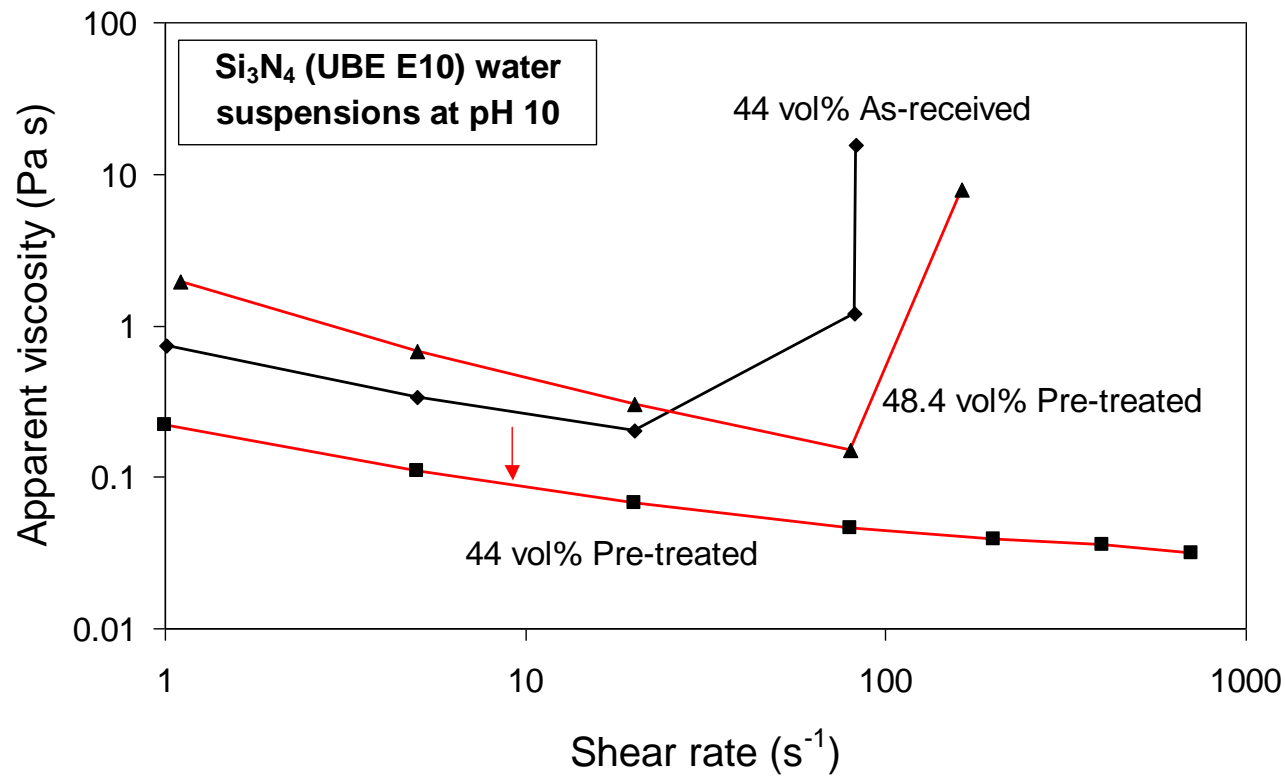
# Re-dispersibility of submicron powders - 1

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

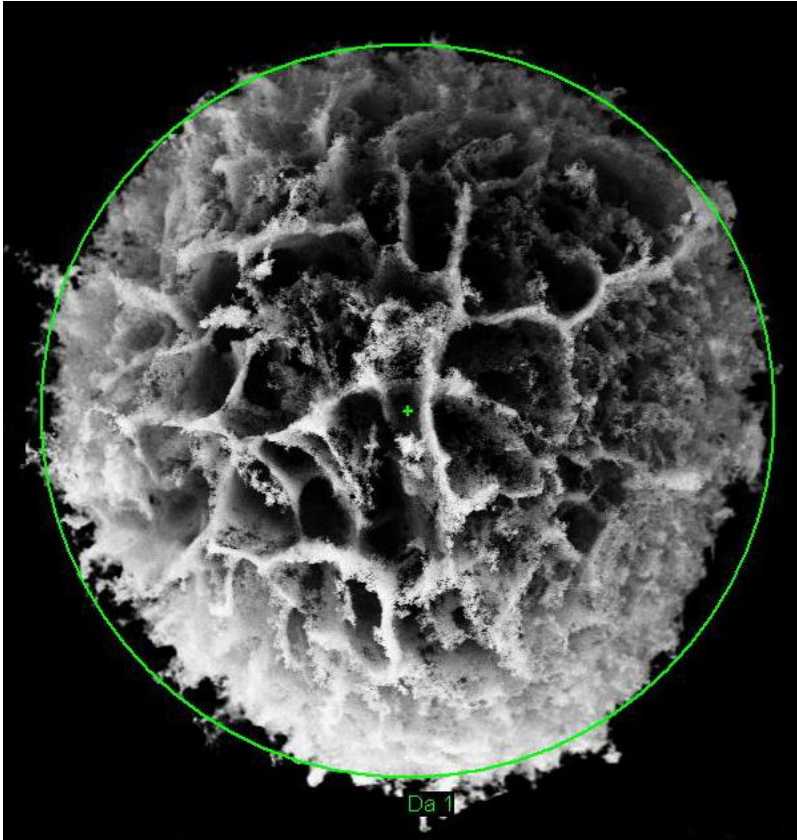


# Re-dispersibility of submicron powders - 2

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



# Granules of "nano" powders



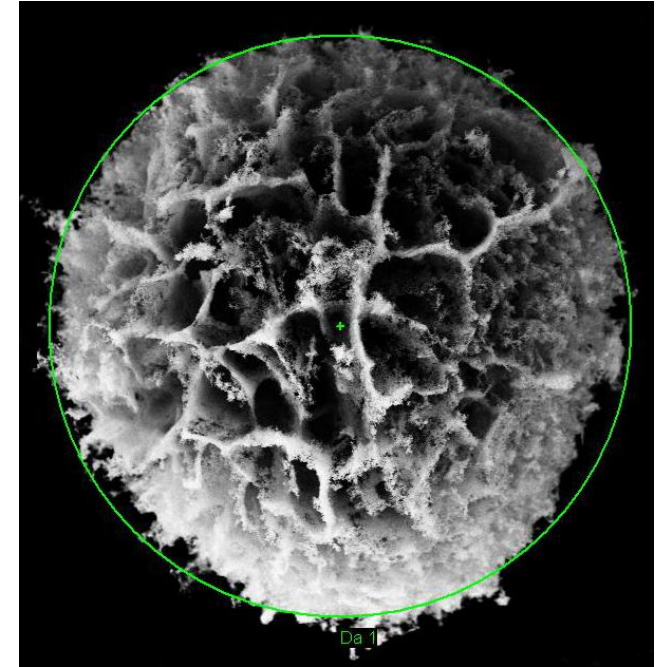
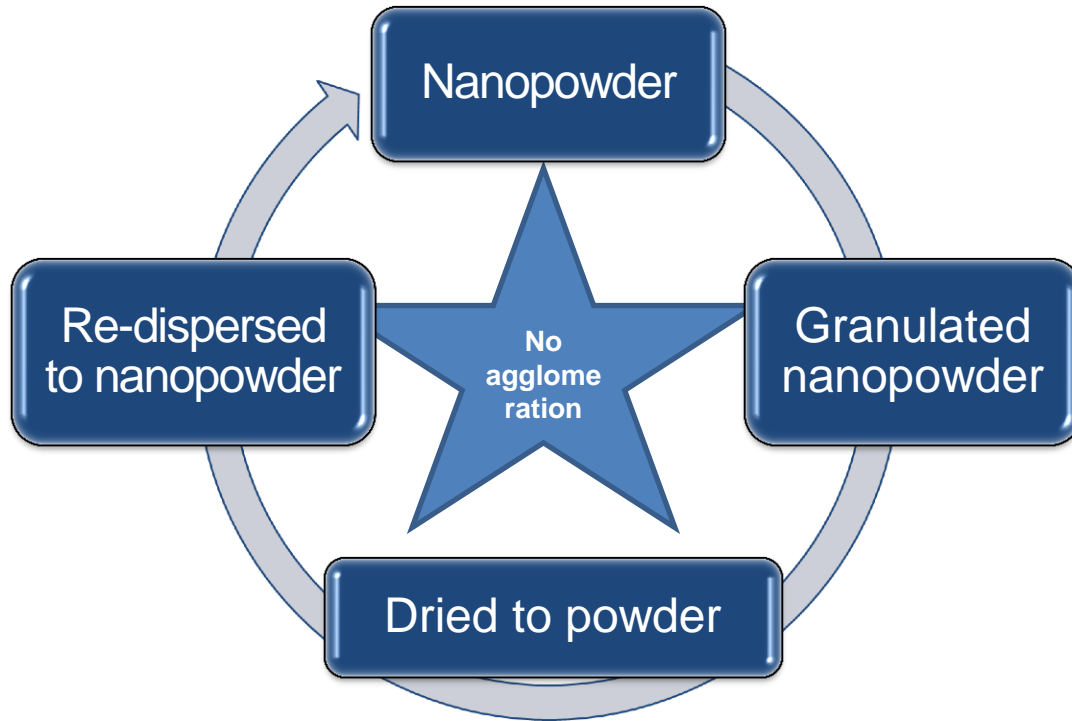
Granule based on 10-30 nm particles at low concentration



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