Freeze Granulation

The Way to Optimised Powder Processing

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Outline

- **Background**
- **History**
- **Freeze Granulation Procedure**
- **Principle of Freeze Granulation (FG) vs Spray-Drying (SPD)**
- **Granule properties**
- **Advantages of FG**
- **Disadvantages of FG**
- **Laboratory Granulator LS-2**
- **Applications**
  - Sintering of pressed SPD vs FG powder
  - Low-dust powder – easy to disperse
  - Enabling mild slip preparation route
  - Medium for unique two-step combustion
- **Summary – Developments**
- **References**

Background

- Pressing is the dominating shaping technique for ceramics and powder metals. Submicron and/or composite powders require adequate preparation to achieve a pressing performance that ensures dense compaction into a homogeneous state.

- Conventional powder granulation methods (spray-drying, sieve granulation etc) show drawbacks. Migration phenomena cause in-homogeneities, cavities and hard granules that do not disintegrate properly during pressing.

- The preparation of a powder suspension by applying colloidal processing combined with sufficient mechanical treatment provides optimal homogeneity.

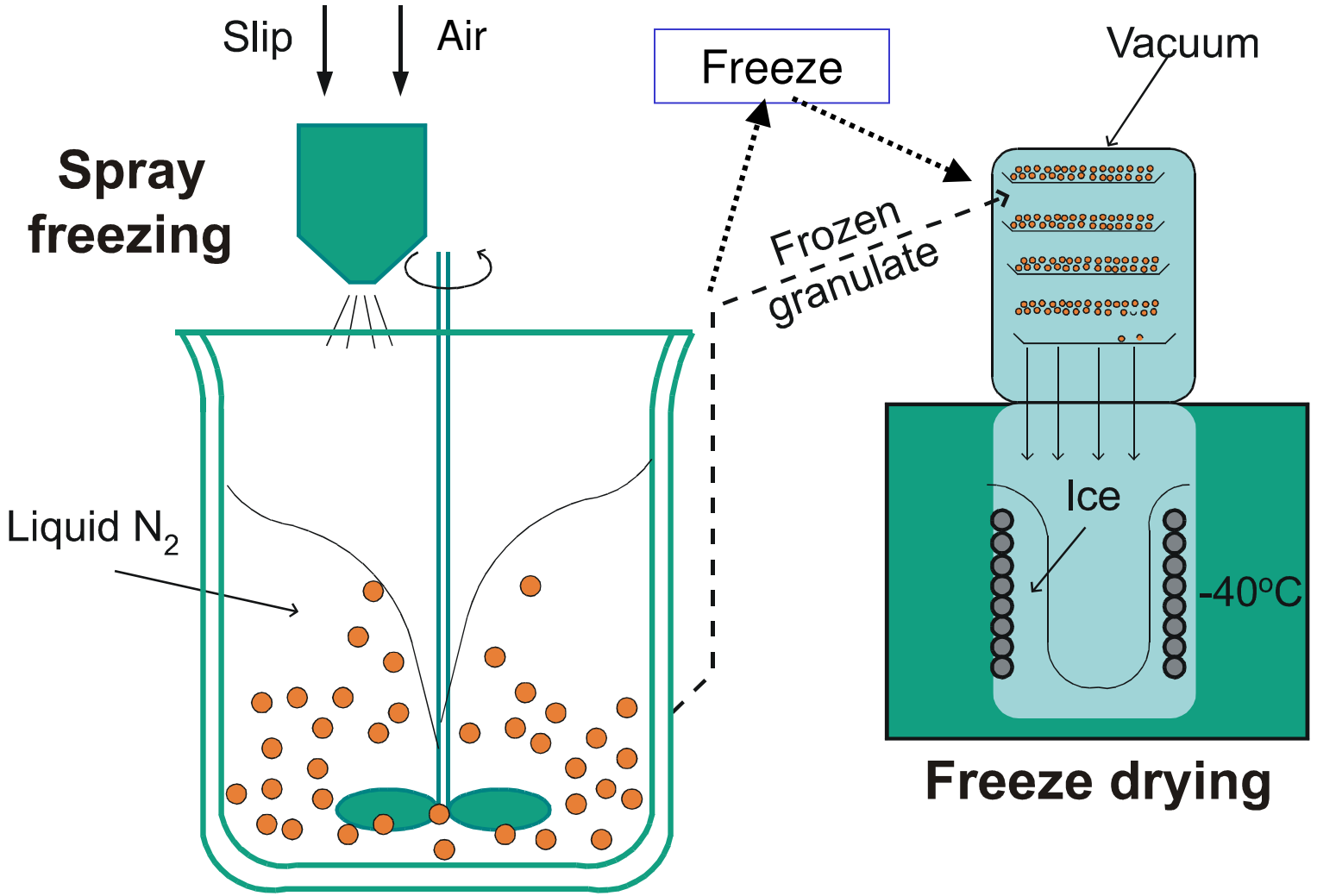
$\Rightarrow$ can be preserved by freezing and subsequent freeze-drying

History

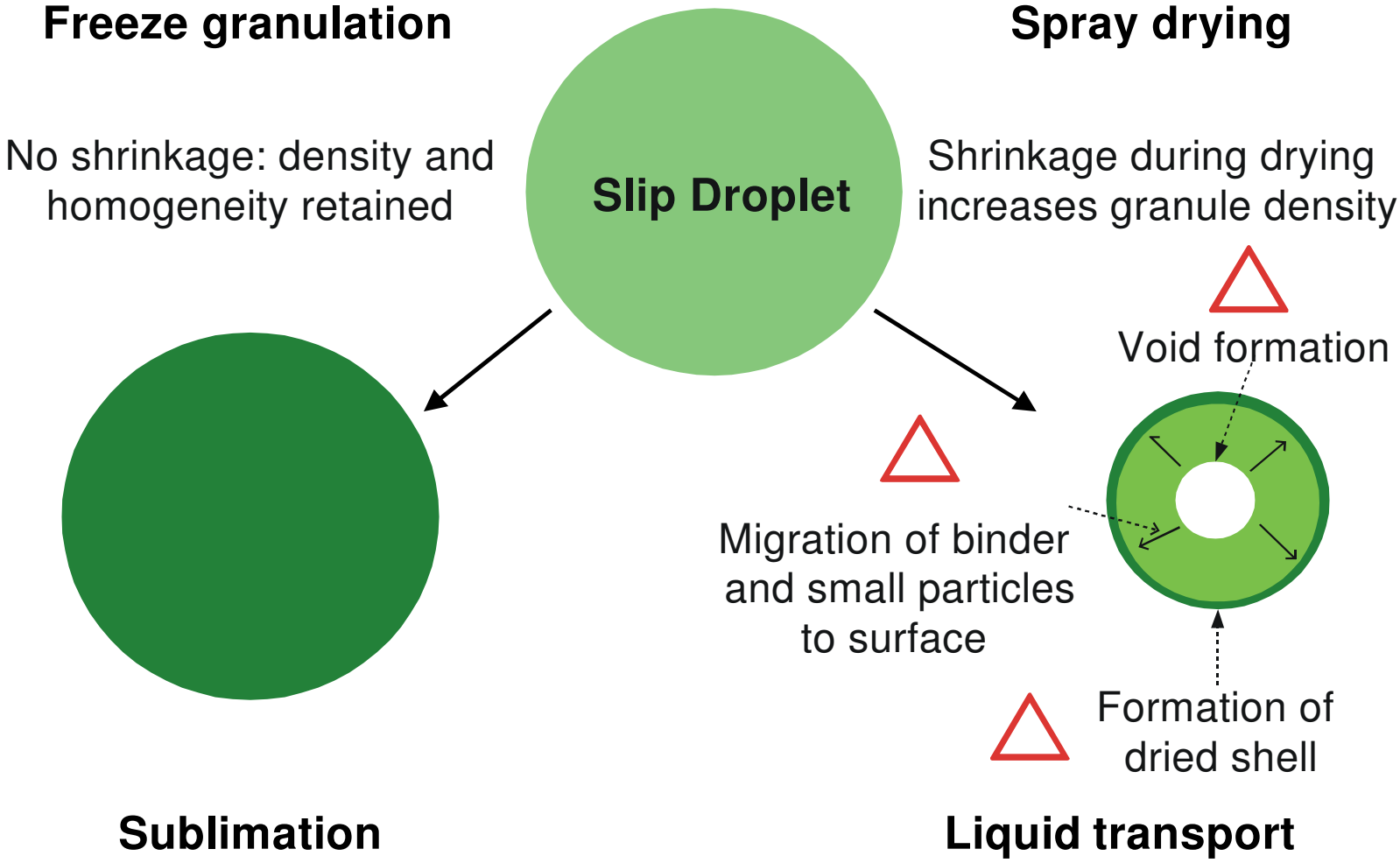
- In mid 70th W.W. Rhodes and S. Prochazka (1975) provided the first publications about freeze granulation of ceramic powders without defining the advantage or explaining the positive results
- Freeze granulation was developed at SCI (now integrated with **IVF**) in the late 1980s as the most suitable method to provide optimal granule properties for lab and research purposes.
- During the 1990s, freeze granulation was successfully used in material and processing developments within many research projects and contract work.

  Specific powder systems from various companies were tested with very promising results. However, no commercial equipment was available and therefore PowderPro AB as a spin-off company was founded in 2000.
- **PowderPro AB** supplies granulation equipment and carries out test granulations (standard concept), whereas **IVF** conducts slip and granulation development.
  - Several companies and research labs around the world have applied the freeze granulation process with the support of IVF/PowderPro.

Freeze Granulation Procedure

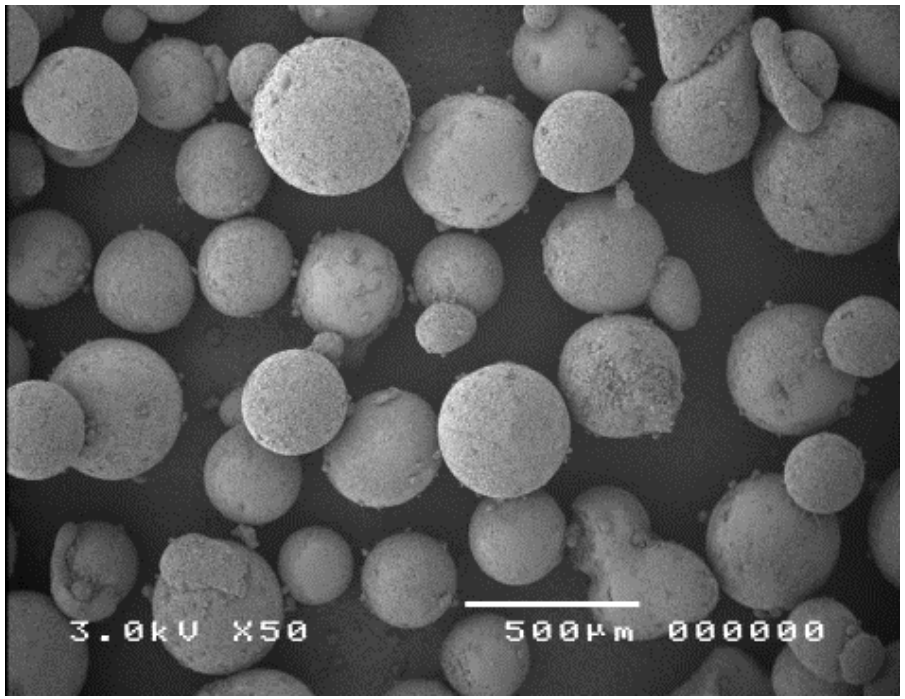


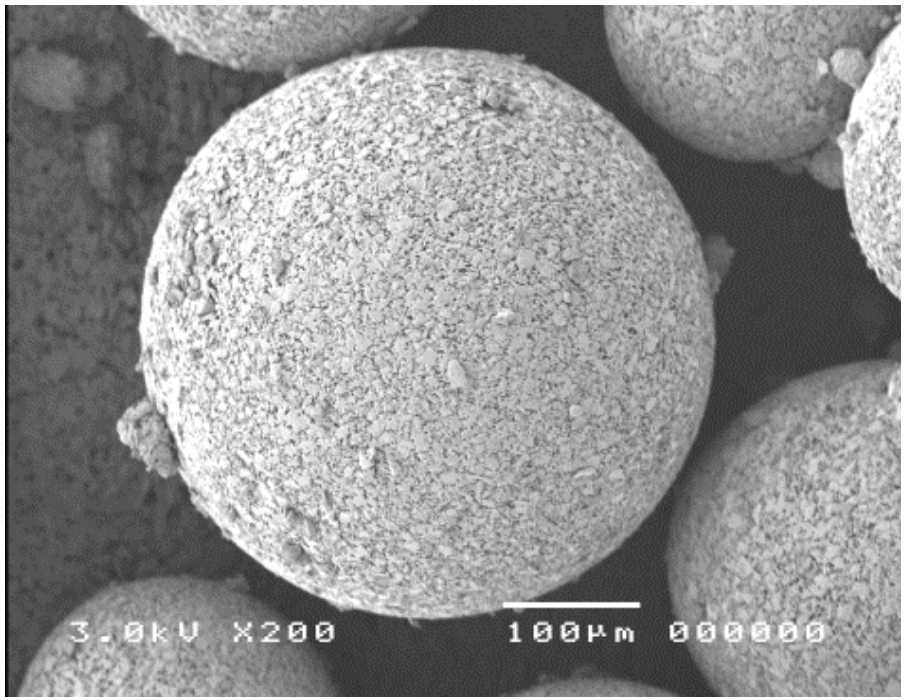
Freeze Granulation vs Spray Drying



Granule Properties

Spray-frozen and freeze-dried granules of a composite material





Free-flowing granules with a wide granule size distribution (50–500 µm). Lower granule and tap density vs spray-dried granules.

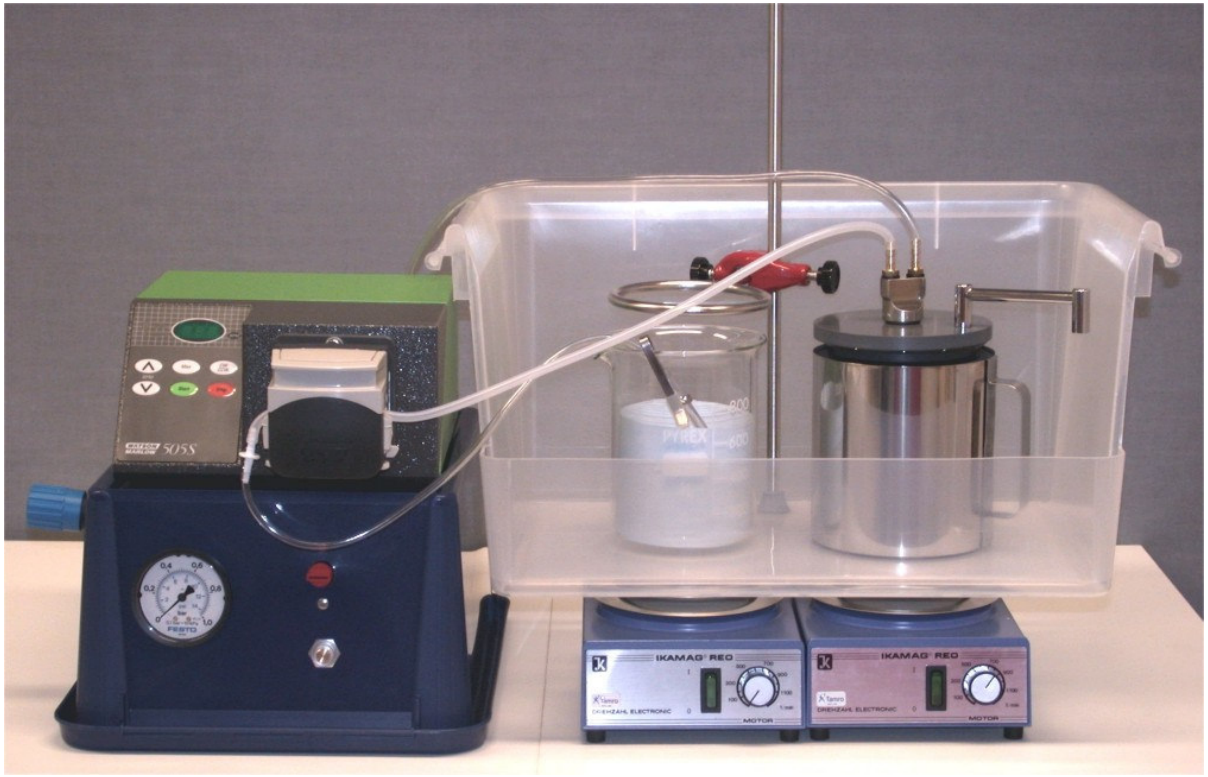
Advantages of Freeze Granulation

- The granule density can be controlled (solid content of the slip)
- No cavities in the granules
- No migration of small particles and/or binder gives a high degree of granule homogeneity
- A mild drying procedure minimises oxidation of non-oxides and metals
- Lower granule density and evenly distributed pressing aids gives softer granules and ensure that all granules are broken during compaction
- Low waste of material (high yield)
- Not only large quantities but also very small experimental batches (50–100 ml slip) can easily be processed
- The equipment is easy to clean– (allows the use of latex as binder)
- Possibility of recycling organic solvents

Disadvantages of Freeze Granulation

- Two process steps: freezing and batch-wise freeze-drying
- Limitation in the choice of solvent, based on the freezing properties – a freezing point between –20 and +10 °C is recommended
- Large-scale equipment with a capacity beyond 30-40 kg granules (corresponds to granulation of ca 25-30 litre ceramic suspension) per day needs to be further developed

Laboratory Granulator LS-2 and LS-6



Capacity to granulate up to 6 l powder suspension per hour

- Easy to set up and clean
- Freeze-dryer with suitable capacity is required

PowderPro Göteborg AB

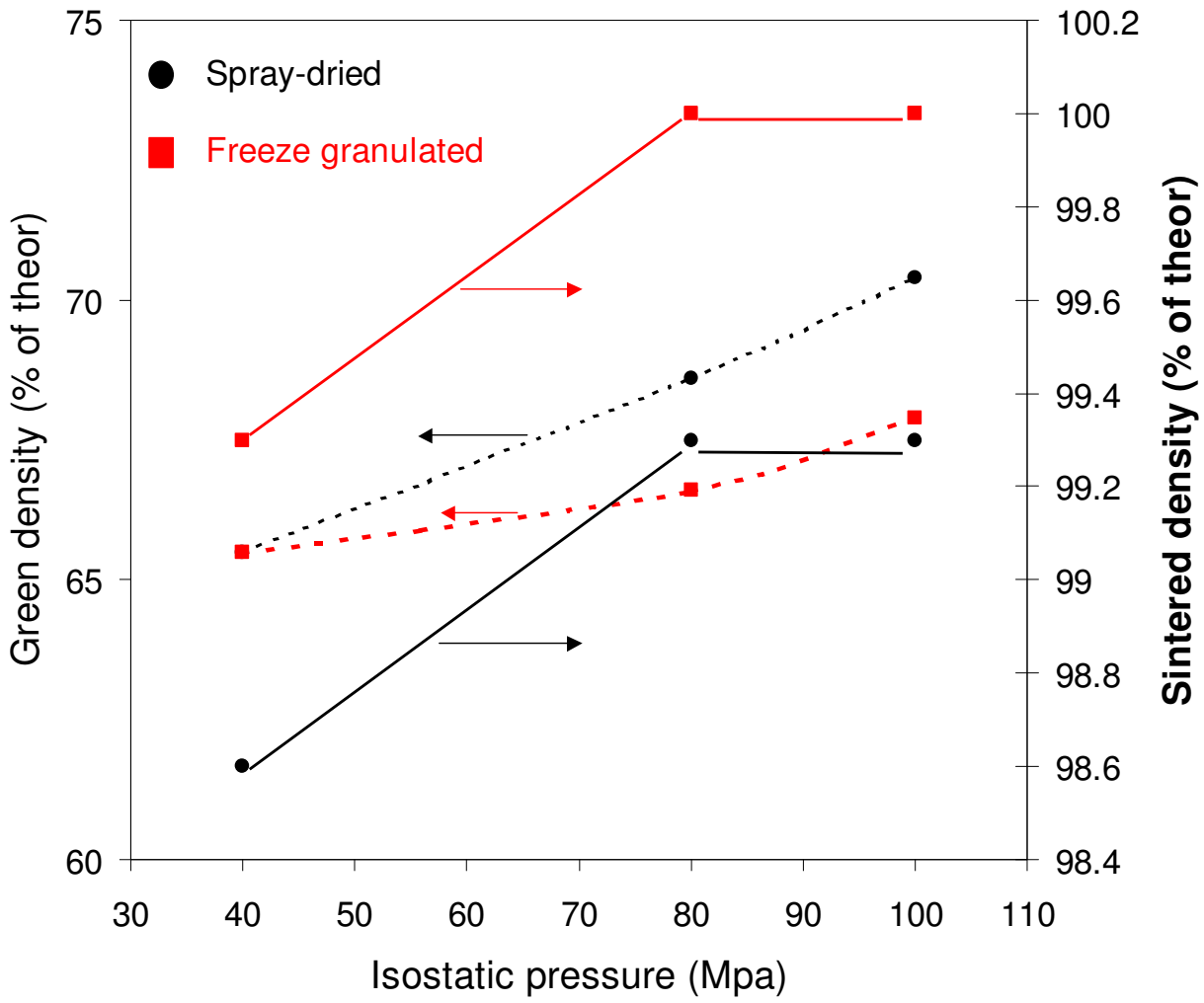
Freeze drier GT-2

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

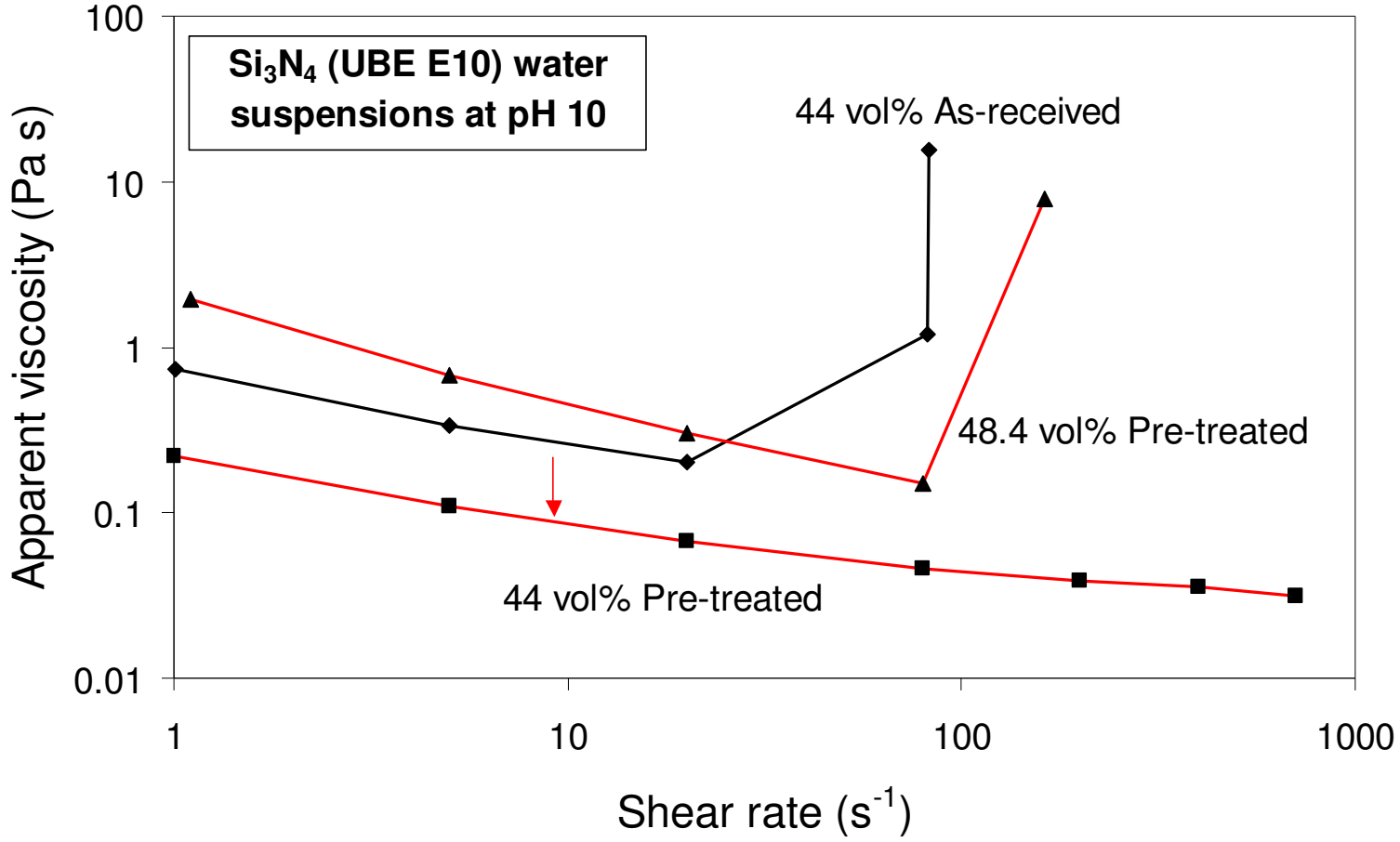
Spray-dried (SPD) vs Freeze-granulated (FG) High Alumina Powder



FG requires lower CIP pressure and gives higher sintered density from the same pressed density than SPD does

Low-Dust Powder – Easy to Disperse

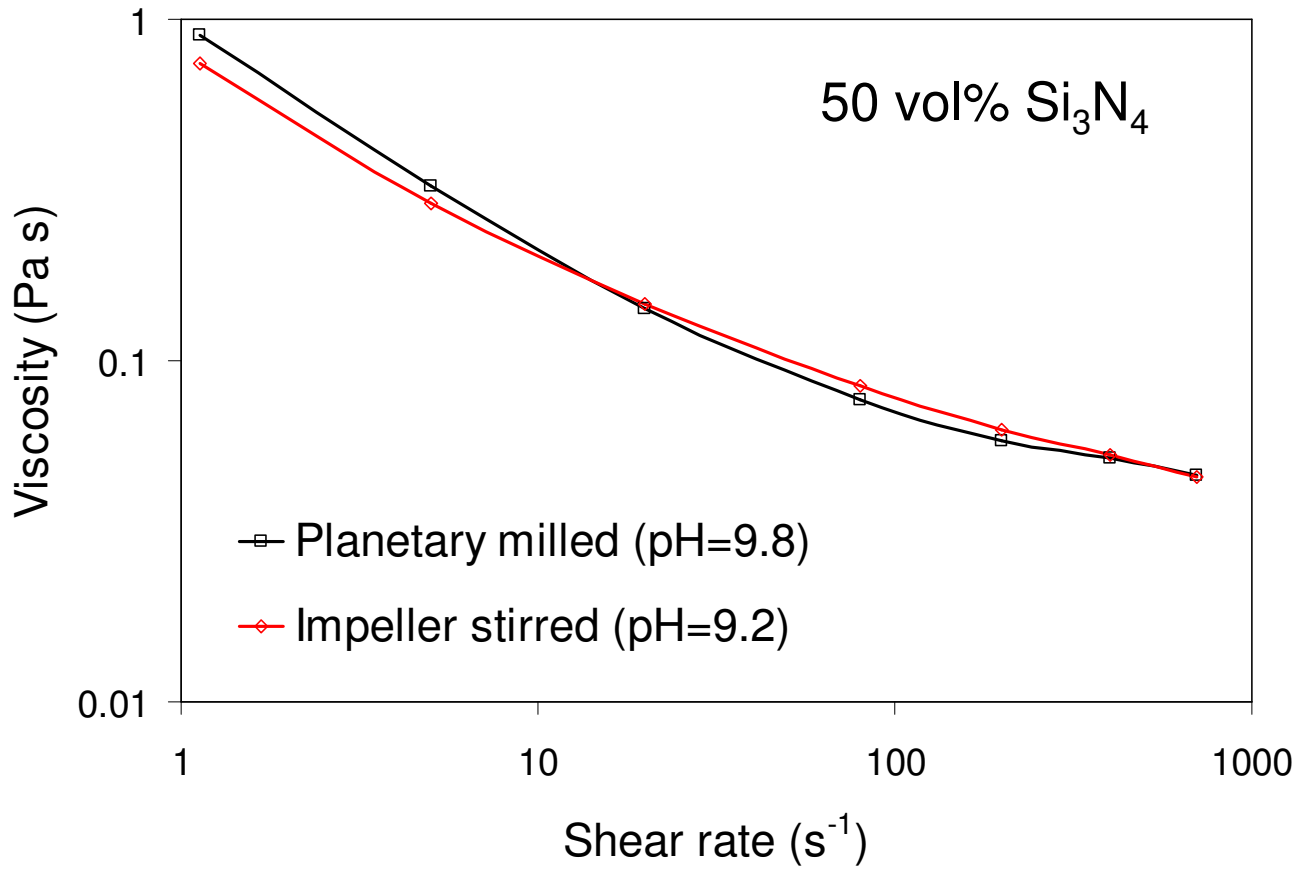
Powders that are difficult to deagglomerate can be dispersed at low concentrations, freeze granulated/freeze-dried and re-dispersed at high concentrations



Dispersion of nano powders can be enhanced!

Slip Preparation under Mild Conditions

Freeze-granulated/freeze-dried submicron powders can be dispersed with impeller stirring to the same level as that of planetary milling



Medium for Two-Step Combustion

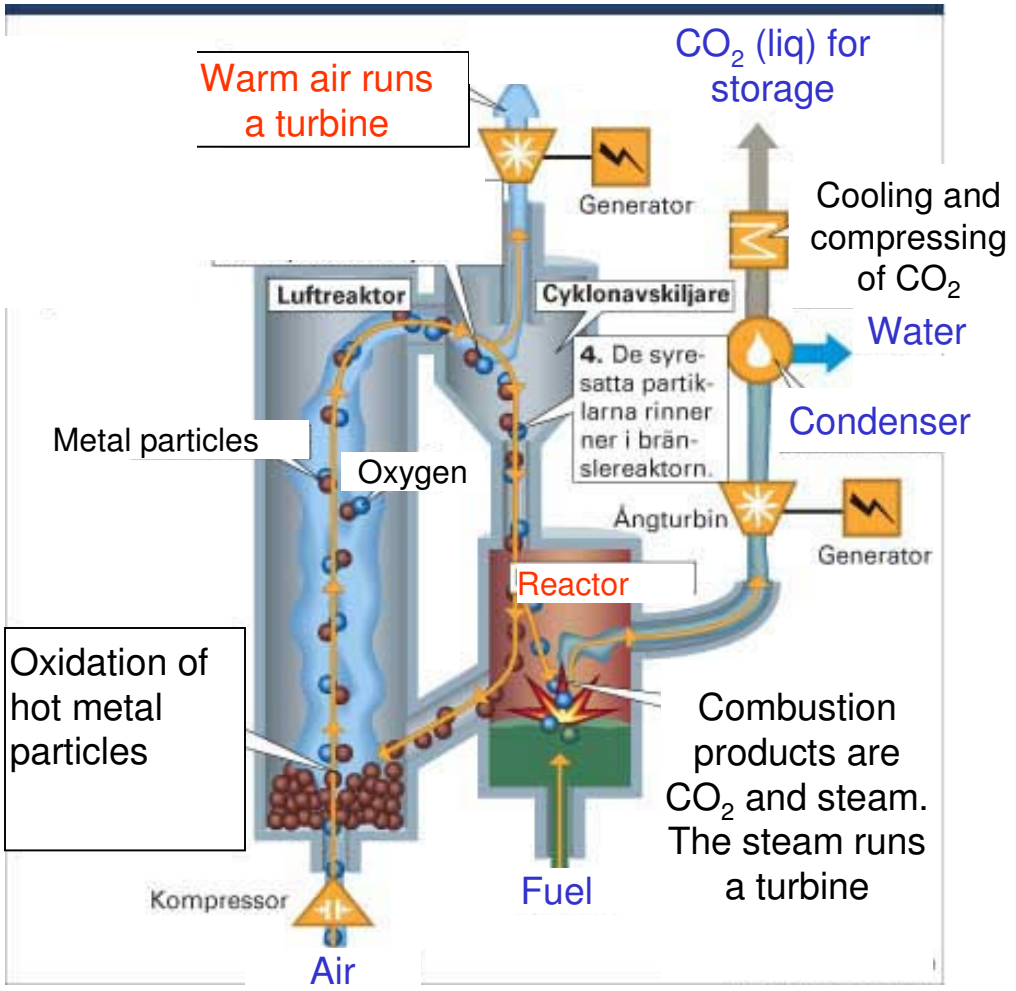
(Department of Energy Technology, Chalmers University of Technology)

- Particles (90–200 µm) of metal oxides catalyse combustion of natural gas via reduction to metal – the metal particles are re-oxidised by air and the process is restarted

  The exhaust gases; water vapour is condensed and carbon dioxide can be stored

- A lot of powder mixes of metal oxides have been freeze granulated, sintered and evaluated regarding reactivity and granule (particle) strength

⇒ The most promising materials have been processed in a 10 kW pilot plant with excellent results



Summary and Developments

- Freeze granulation has shown to be a competitive technique for the manufacture of granules for pressing owing to its ability to preserve the material homogeneity and enhance the pressing performance
  - Small granule quantities as well as larger ones can be produced with equal properties
  - The granule density can be controlled by the solid content of the powder suspension to be granulated
  - The mild drying provides a low degree of oxidation of non-oxide ceramics or metals and gives soft granules that are easily disintegrated during pressing or at redispersing to a suspension
- In pipe-line
  - Technical development of the first large-scale freeze granulator
  - Optimised system for processing in organic media

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