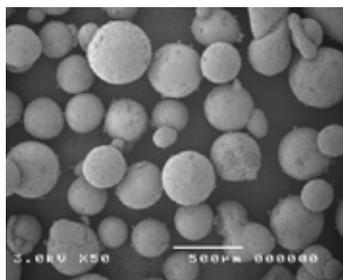


# Freeze Granulation

## Homogeneous powder granules with superior performance

Manufacture of ceramic components by powder pressing most often requires a granulation operation prior to pressing. The aim is to obtain free flowing granules that evenly fill the pressing die and easily disintegrate at pressing. Granulation is normally conducted by an initial mixing of the powder and appropriate pressing aids (binder, plasticiser) in a liquid. The suspension can then be dried with some remaining liquid and sieve granulated (small scale) or spray-dried (large scale). However, there are drawbacks with these granulation techniques. Sieve granulation does not result in spherical granules and, therefore, exhibit non-favourable flow properties. Both techniques involve drying in air with liquid transport within the granules that creates a significant risk for segregation of binder and/or small particles to the periphery. The result will be inhomogeneous and hard granules that do not disintegrated properly at pressing. Ultimately the consequence will be poor material properties.



Granules of a composite material produced by freeze granulation.

SCI has adopted and developed an alternative technique – freeze granulation (FG) – which enable preservation of the homogeneity from suspension to dry granules. By spraying a powder suspension into liquid nitrogen, the drops (granules) are instantaneously frozen. In a subsequent freeze-drying the granules are dried by sublimation of the ice without any segregation effects as in the case of conventional drying in air. The result will be spherical, free flowing granules, with optimal homogeneity. FG provides optimised condition for the subsequent processing of the granules, for example easy crushing to homo-

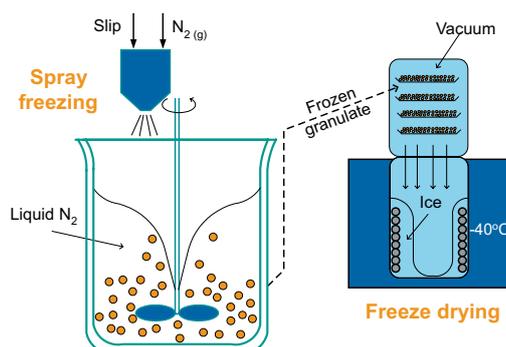
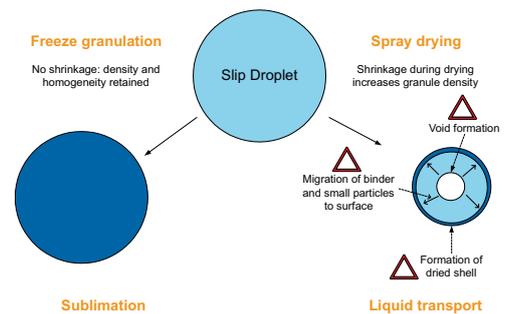


Illustration of the freeze-granulation process.



Comparison of drying effects in spray-drying vs freeze granulation.

geneous and dense powder compacts in a pressing operation. High degree of compact homogeneity will then support the following sintering with minimal risks for granule defects. Besides, high degree of granule homogeneity, FG offers several other advantages:

- ▶ Control of granule density by the solids content of the suspension.
- ▶ Mild drying prevents serious oxidation of non-oxides and metals.
- ▶ No cavities in the granules.
- ▶ Low material waste (high yield).
- ▶ Small (50–100 ml suspension) as well as large granule quantities can be produced to equal quality.
- ▶ Easy clean of the equipment (latex binder can be used).
- ▶ Possibility to recycle organic solvents.

The granule size distribution will be controlled by the suspension rheology (flow properties) and the process parameters (pump speed and air pressure). Normally, a certain size distribution-width is achieved with an average size, typically, around 100–200 µm. In a comparison between the sintering performance of pressed specimens using granules obtained by FG or spray-drying (SD) the effect of homogeneity is clearly illustrated. Low pressure at CIPing results in the same compaction but a significant higher sintered density for FG-specimens. Easy crushable granules and high degree of homogeneity is re-

## Contact us for more information

Do not hesitate to contact us if you want to have more information or have specific questions that you want to discuss. Based on your requests and needs we can provide a quotation.

More information is provided by:

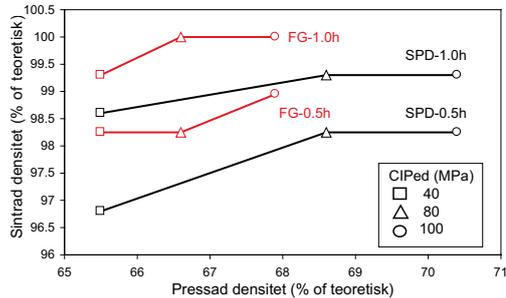
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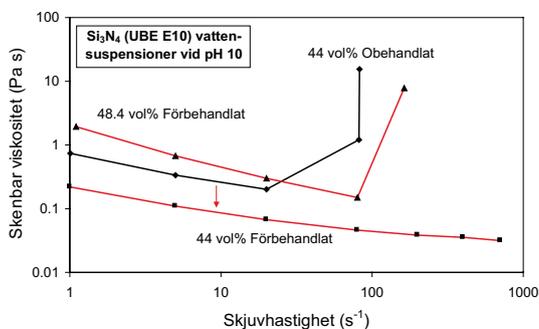


CIPed and sintered alumina specimens form spray-dried (SD) or freeze granulated (FG) powder.

sponsible for this. Higher CIP pressure gives clearly higher compact density for SD that support the sintered density but still FG shows its superior performance by equal or higher degree of densification.

## Other uses of high performance powder granules

Although, the main purpose of the powder system for freeze granulation has been to obtain granules with high performance in pressing, other interesting applications for the technique exist. One such example is to produce metal oxide particles as a catalytic active material in combustion facilities.



Suspension viscosity with pre-treated (freeze granulated/freeze dried) and as-received  $\text{Si}_3\text{N}_4$  powder.

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Freeze granulation in combination with the mild drying procedure can also be used to produce low-dust powders that easily can be re-dispersed. This is of special importance when fine powders, for example nano-powders, are to be processed. By an easy break down of the granules it will be easier to reach a high particle concentration with a low energy input.

## Services and Equipment

We have built up considerable experiences and know-how about freeze-granulation within a wide range of ceramic, metallic and composite materials. The industrial interest of freeze granulation has continuously grown and recently been intensified. Based on promising tests and developments, successful commercial applications have been established. This has also been possible as equipment for freeze granulation recently became commercial available.

We provide help in optimising suspension properties, the granulation procedure as well as to characterise granule properties and conduct pressing studies. Small batches of granules for technique evaluations as well as small to medium size production volumes can be produced. We can also provide support regarding equipment and production set-up.



Lab-scale freeze granulator (LS-2, PowderPro AB)

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