CERAMIC AND COLLOIDAL PROCESSING - EXERCISES

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Exercises 11

1. **What is the preferred shaping (forming) method for fabricating the following parts and why is the method better suited than others?**

- a spherical part of high quality (for example a hip prosthesis)
- a multilayer capacitor (for example - barium titanate ceramic)
- a toilet (porcelain)
- a ceramic knife (in alumina simple geometric shape)
- a static mixer (very complex shape)
- a red brick

Solution.

Preferred method of shaping to manufacture:

-a spherical part of high quality: cold isostatic (dry) pressing – homogeneous green density
-a multilayer capacitor: tape casting – best method to produce thick films difficult to dry press or slip cast
- a toilet: slip casting – very large objects with complex shape not possible to dry press or extrude
-a ceramic knife: uni-axial dry pressing – thin (i.e. very low aspect ratio) so good pressure distribution, rapid and automatic
-a static mixer: injection molding (or deposition by computer-assisted layers) – very complex shape – not possible by dry pressing or even slip casting as parts have orthogonal directions.
- red brick: extrusion – simple 2D shape e.g. (https://www.youtube.com/watch?v=GEvoXuFKSA0)

2. **What is the disadvantage of an agglomerated (flocculated or coagulated) suspension for shaping a raw body by a wet process? Could you find a case where it can be beneficial?**

Solution.

The main drawback is that aggregates sediment faster, which can make processing difficult and leads to an inhomogeneous packing and large pores in the green body. In addition, the porosity in the raw body is also increased due to the porosity in the agglomerate. These lead to inhomogeneous shrinkage during the sintering step and often leave large pores which reduce the strength and general properties of the ceramic.

However, flocculation (agglomeration by a polymer at low surface coverages such that the polymer can attach to both particle surfaces) or coagulation (agglomeration by increasing the salt or ionic concentration of the dispersing liquid that compresses the electrical double layer) makes it possible to limit possible size segregation (and / or composition in a mixture) of particles. The pores created are also more interconnected, which facilitates filtration and makes filter pressing faster. As long as the porosity is uniform and of similar size sintering can still lead to high quality ceramics.

3. **What type of law as a function of time does the thickness of a slip-casting or filter-pressing part follow? What are the practical consequences? How long is necessary to form a thickness of 5 and 10 mm of an alumina slip by filter pressing with an overpressure of 10 bar ($Kp = 3\ 10^{-16}$ m^2, d_{sv} = 0.3 μm, v_1 = 0.64 v_0 = 0.3, η = 3 mPa,)**

Solution.

The thickness of the consolidated layer in slip casting or filter pressing follows a parabolic law:

$$d = \sqrt{\frac{2Kp \cdot Pt}{2}\left(\frac{v_1}{v_0} - 1\right)}$$

with $Kp=3\cdot10^{-16}m^2$, permeability of the deposited ceramic layer

$P=10^6$ Pa, hydrostatic pressure

$$\rightarrow t = \frac{d^2\eta}{2Kp \cdot P\left(\frac{v_1}{v_0} - 1\right)}$$

$\eta=3\cdot10^{-3}$Pas

t(d=5mm)=110 secs

t(d=10mm)=440 secs

V_1= 0.64 volume fraction of solids in the consolidate ceramic layer

V_0= 0.3 volume fraction of solids in the suspension

4. **What kind of commercial powder do we use for dry pressing in industry? How are these powders produced?**

Solution

The powders used for pressing for dry pressing are always granulated (dv50 = 50-300µm) normally by atomization (Spray Drying). This ensures good flowability of the powder and good filling of the die, giving accurate dimensional stability of up to a few microns after the sintering step.

5. **What are the steps in the manufacture of a ceramic part after shaping (forming) and before sintering? What are the problems that can be encountered in these steps and how can they be avoided ?**

Solution

There are two important and time-consuming stages:
Drying and Binder Removal (Binder Burnout):

<u>Drying:</u>
Shrinkage of the ceramic part due to the removal of liquid through the green body porous network can cause stresses if not done in a consistent and controlled manner. If done too quickly, inhomogeneous drying fronts can create pressure gradients and hence internal stresses which can cause cracks after the critical point when the liquid meniscus enters the ceramic green body. Such gradients can be minimized - with slow drying (temperature not too high or in an atmosphere with high relative humidity> 60%).

<u>Binder removal or Binder burnout:</u>
The most popular is the pyrolytic route (Binder burnout): one must be careful not to be in an oxidizing atmosphere if the ceramic is not an oxide. The residues of the polymers used must also be well removed, a small amount of water vapour can sometimes help (i.e. a slightly humid air 10-20% RH). If we form closed pores during the sintering cycle that still contain carbonaceous residues, gas pressure (from the

reaction of the carbon to form CO/CO2 can swell and crack the part in the sintering cycle at these high temperatures > 1000°C. Consequently, the kinetics of evaporation or decomposition of the organic additives must not exceed the diffusion rate of the species escaping from the ceramic green body otherwise pressure gradients appear and the green part may crack. Other methods include capillary flow for wax-based binders, where we go above the melting point and the liquid flows out of the sample. Also, it can be carried out by solvent extraction (simple solvent or using supercritical fluids such as CO_2). The particles can however rearrange which can induce deformation of the part especially if all binders are removed at the same time i.e. all the binders must be soluble in this solvent. Sometimes mixtures of binders are used such that extraction kinetics for each are different allowing for small rearrangements without significant and inhomogeneous deformation.