

# FREEZE FOAMING - TOWARDS CELLULAR STRUCTURES FOR VERSATILE APPLICATION

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## **MOTIVATION**

In addition to the typical properties of ceramics such as wear resistance or brittleness, porous cellular ceramics are characterized by a broad variety of possible applications. Potential applications range **from biomedicine** (membrane bioreactors, bone scaffolds), through biomimetics (honeycomb filters) **to mechanical and plant engineering** (reactors, burners, insulators and refractories). The presented freeze-foaming process has the potential to cover at least two of these fields.

Freeze-foaming that is foaming of an aqueous ceramic or metallic suspension with subsequent freeze-drying to a porous, cellular structure.

### **EXPERIMENTAL**

Ceramic starting material for biomedicine: Hydroxyapatite, HAp (SIGMA-ALDRICH),  $d_{50}$ :2.04 µm, BET: 72.7 m²/g or ZrO<sub>2</sub> (TOSOH), TZ3Y-E,  $d_{50}$ : 0.73, BET: 6.9 m²/g

**Ceramic starting material for refractories**: mullite (NABALTEC), bimodal mixture (K0/K0c) with a  $d_{50}$ :10.4 µm, BET: 1.37 m<sup>2</sup>/g.

Organic additives: 2-10 resp. Vol.-% dispersant agent and thickener. Powder Processing: The ceramic powders and organics are dispersed in water by a stirrer or a kneader depending on the processing viscosity. Foaming and Shaping: The homogenized suspension is cast into a synthetic rubber mold and then transferred to a freeze drying device (Co. CHRIST, Model 1-20). By reducing the ambient pressure the suspension inflates. Further pressure reduction is accompanied by a sudden freezing of the foam and the subsequent freeze drying to a stable porous structure.

### RESULTS

After debinding and sintering, depending on the starting material, a bioac-

It could be proved that the bioactive HAp foam is suited for cell cultivation and even differentiation (figure 3 and 4). If toughness values similar to those of a real bone are achieved, this ceramic foam might be used as implant.





Fig. 3: Live staining in the pores of the HAp foam

Fig. 4: Collagen 1 proof (21 days) in the well around the HAp- $ZrO_2$  foam

In further studies, the freeze-foaming technique was successfully used to upscale the manufactured foams to bigger geometries. Using a mullite powder a freeze foamed structure (235 x 114 x 70 mm<sup>3</sup>) was achieved which might be used as lightweight insulating brick in the refractory industry (figure 5). As shown in the lower diagram, the first tested samples (H01-H09) have already met the necessary requirements of refractories. The determined thermal conductivity is similar to state-of-the-art products (figure 6).



tive HAp foam and a mullite lightweight brick were achieved using the freeze-foaming technique. By using a specific, outer shape, for example, synthetic rubber one can achieve the porous counterpart (figure 1 and 2).



Fig. 1 HAp thumb bone replica via freeze foaming

Fig. 2 Open and interconnected , porous freeze-foamed HAp structure

Fig. 5: Lightweight insulating brick (H06)

Fig. 6: Comparison of thermal conductivity values of freeze-foamed mullite samples with the lightweight brick Porrath FL 30-11 (RATH AG)

#### CONCLUSION

Freeze-foamed cellular structures successfully have been manufactured for completely different applications. So far, only in laboratory scale, however, through a specific adjustment of process parameters and equipment, the manufactured quantity & quality can be increased. In addition to the presented starting material, other ceramic powders (like SiC, ZrO2, Al2O3, hybrids) and also powder metals (e.g. steel) can be created to porous parts.