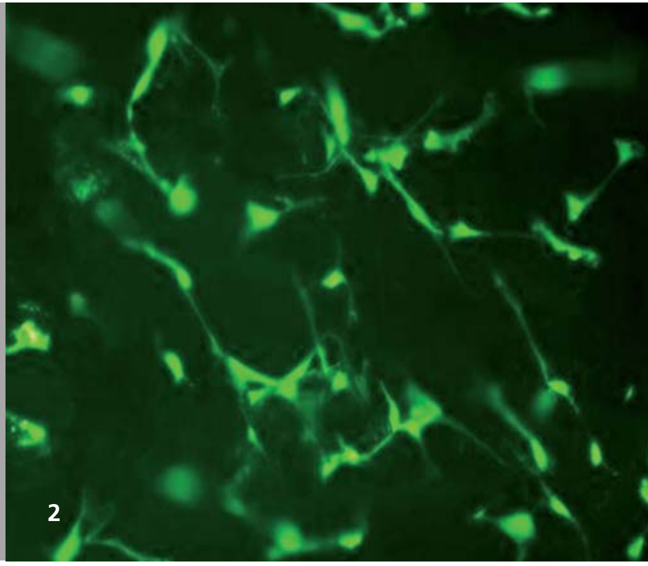

FREEZE FOAMING – CELLULAR STRUCTURES FOR VERSATILE APPLICATIONS

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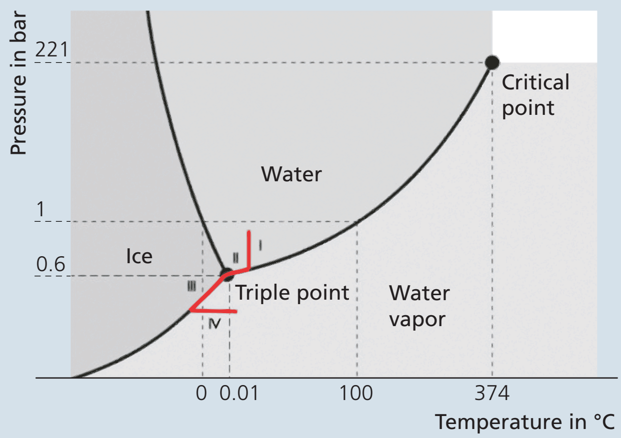
In addition to the typical properties of ceramics such as wear resistance or brittleness, porous cellular ceramics are characterized by a broad variety of possible applications. Potential applications range from biomedicine (membrane bioreactors, bone scaffolds), through biomimetics (honeycomb filters) to mechanical and plant engineering (reactors, burners, insulators and refractories).

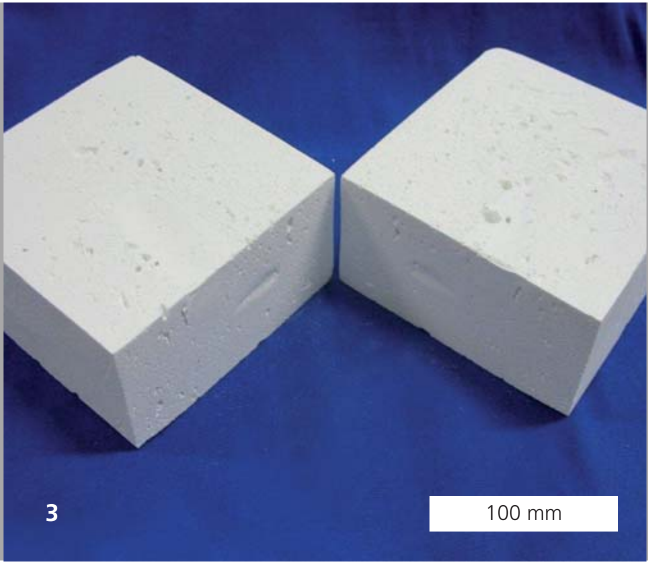
The presented freeze-foaming process has the potential to cover at least two of these fields. Freeze-foaming that is foaming of an aqueous ceramic or metallic suspension with subsequent freeze-drying to a porous, cellular structure. For this purpose, the ambient pressure is reduced in a freeze-drying device through which water vapor and residual procedural air inflates the suspension (diagram on the right, curve sections I, II). As evaporation in aqueous suspensions causes a drop of temperature, the foaming suspension suddenly freezes at the intersection of the liquid-vapor-solid equilibrium line (triple point). By heating the panels underneath, the now stable protofoam is freeze-dried due to sublimation of the frozen water (curve section IV).

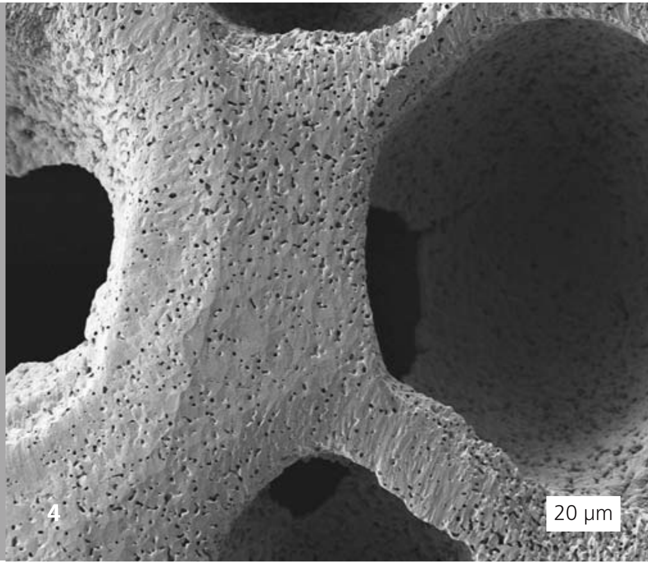
Thus, a porous, biocompatible ceramic foam was manufactured using different hydroxyapatite powders (Merck KGaA and SIGMA-ALDRICH Cooperation). It could be proved that this foam is suited for cell cultivation and even differentiation (figure 2). If toughness values similar to those of a real bone were achieved, the ceramic foam might be used as implant. The freeze-foaming technique also provides the possibility to manufacture a specific, outer shape of a porous foam by using, for example, synthetic rubber as counterpart. Figure 1 shows such a hydroxyapatite foam replica in shape of a human thumb bone. The pore structure fulfilling the requirements to cultivate cells can be characterized by non-destructive X-ray computed tomography (PROCON X-Ray, max. 150 kV) and image analyzing software (Porescan, Image J) as well as mercury porosimetry.

In further studies, the freeze-foaming technique was successfully used to up-scale the manufactured foams to bigger geometries. Using a mullite powder (NABALTEC, K0/K0c) a freeze foamed structure (235 x 114 x 70 mm³) was achieved which might be used as light-weight insulating brick in refractory industry (figure 3). As shown in the lower diagram, the first tested samples (H01-H09) have already met the necessary

p-T diagram of water; course of freeze foaming process



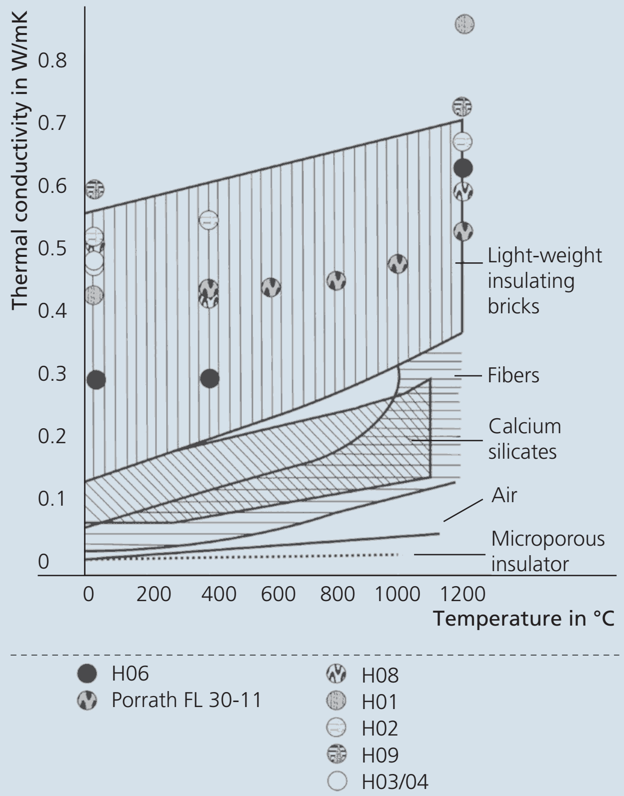




requirements of refractories. The determined thermal conductivity is similar to state-of-the-art products.

So far, freeze foamed cellular structures have been manufactured in laboratory scale. However, through a specific adjustment of the process parameter and the experimental equipment, the manufactured quantity can be increased. In addition to the presented starting material, other ceramic powders (like SiC, ZrO_2, Al_2O_3, hybrids) and also powder metals (e.g. steel) can be transferred to porous semifinished parts.

Comparison of thermal conductivity values of freeze-foamed mullite samples with products of Rath AG (Porrath FL 30-11)



Services offered

- Freeze foaming
- Freeze drying
- Development of suspensions

1 Hydroxyapatite foam replica in shape of a human thumb bone.

2 Vital staining of cultivated human mesenchymal stem cells in the hydroxyapatite foam, proved using fluorescein diacetate, in cooperation with Fraunhofer IBMT.

3 Light-weight insulating brick by freeze foaming.

4 Freeze-foamed microstructure, ion-polished (hydroxyapatite).