

# STRENGTH ENHANCED BIOACTIVE HYDROXYAPATITE FREEZE-FOAMS

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## MOTIVATION

In the course of demographic change lots of research is being conducted to obtain biocompatible materials and implants allowing tissue material ingrowth, osteoconduction and resorption of the artificial material. However, suitable porous ceramics either do not reach the sufficient compressive strength or the approach to such structures is cost-intensive and with high environmental impact. The aim of this research is to achieve a high strength porous bioactive hybrid structure by mixing a bioactive stress sensitive material with a biocompatible stress enhancing material. The so-called freeze-foaming, as a direct-foaming technique and as a new and alternative way to produce cellular structures, is the promising choice for meeting the challenging requirements.

## EXPERIMENTAL

Ceramic starting material: HAp (SIGMA-ALDRICH),  $d_{50}$ : 2.04  $\mu\text{m}$ , BET: 72.7  $\text{m}^2/\text{g}$ ; and  $\text{ZrO}_2$  (TOSO), TZ3Y-E,  $d_{50}$ : 0.73, BET: 6.9  $\text{m}^2/\text{g}$ .

Organic additives: 5 % dispersant agent and thickener.

Powder processing: The ceramic powders and organics are dispersed in water by a stirrer and thoroughly homogenized by an ultrasonic sonotrode.

Foaming and shaping: The 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 (varying temperatures: 10-50  $^{\circ}\text{C}$ ) inflates. Further pressure reduction is accompanied by a sudden freezing of the foam and the subsequent freeze drying to a stable porous structure.

## RESULTS

The freeze-foaming generates interconnected micro-, meso- and macropores as well as open porosity (Figures 1 and 2).

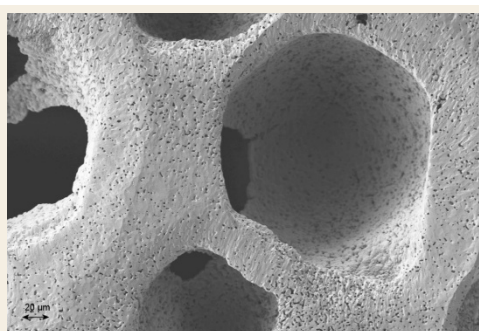


Figure 1: Open and interconnected pores of a freeze-foamed HAp-ZrO<sub>2</sub> structure.

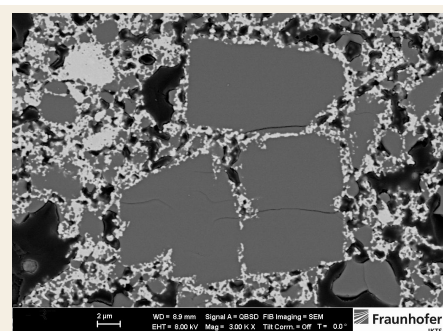


Figure 2: Microstructure of the hybrid foam (grey: HAp, white ZrO<sub>2</sub>).

By using stress enhancing ZrO<sub>2</sub> the compressive strength of a cellular hydroxyapatite is more than tripled (0.6 to 2 MPa). Also, the suspension temperature prior to the foaming process has a high impact on the compressive strength. At low temperatures the resulting strength is thirteen times higher than a pure hydroxyapatite foam (0.6 to 8 MPa) (Figure 3).

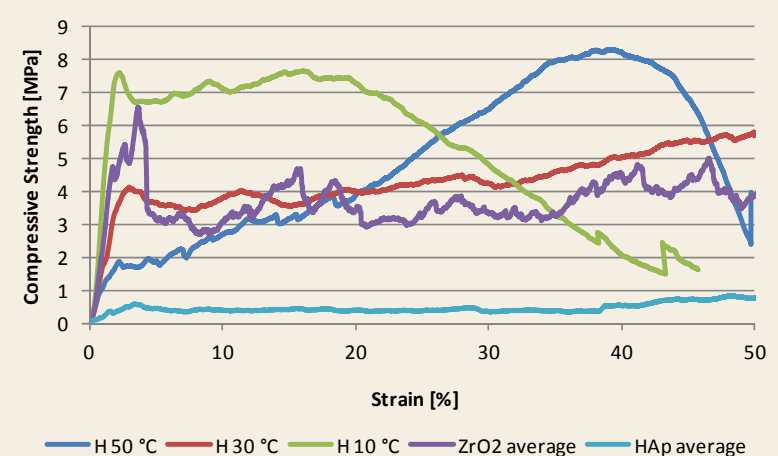


Figure 3: Compressive strength comparison of hybrid (H 10-50  $^{\circ}\text{C}$ ) and pure freeze-foams.

Furthermore, biological experiments with human mesenchymal stem cells (hMSCs) proofed these compressive strength enhanced structures able to allow the ingrowth (Figures 4 and 5) and even differentiation (Figures 6 and 7) of cell tissue material.

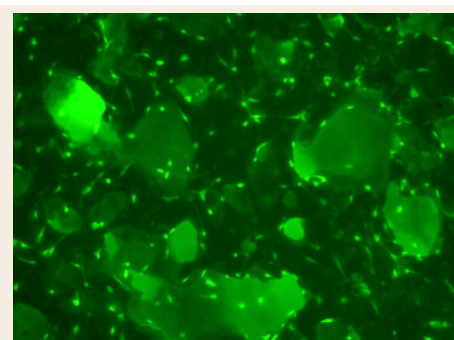


Figure 4: Live staining in the pores of the HAp-ZrO<sub>2</sub> foam (marker: FDA).

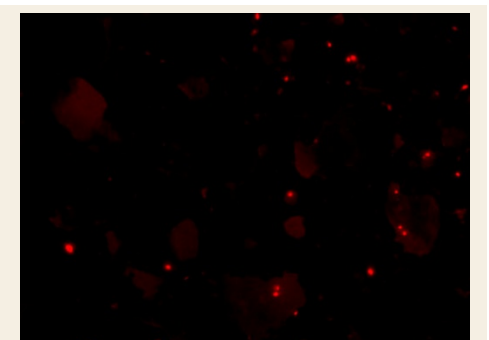


Figure 5: Dead staining in the pores of the HAp-ZrO<sub>2</sub> foam (marker: propidium iodide).



Figure 6: ALP proof in the well (9 days).

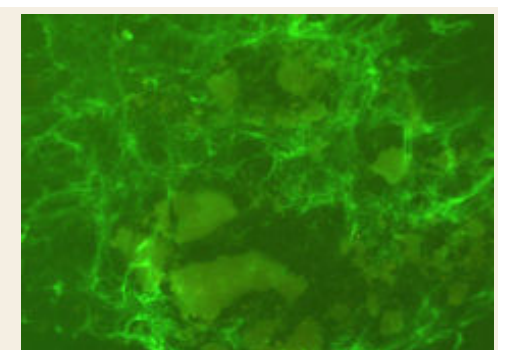


Figure 7: Col.1 proof (21 days) in the well around the HAp-ZrO<sub>2</sub> foam.

## CONCLUSION

Via freeze-foaming a porous HAp-ZrO<sub>2</sub>-hybrid structure was achieved which shows significantly improved compressive strength and good cell ingrowth behavior of human mesenchymal stem cells.