

## Understanding crystalline phase transformations in calcium phosphate bone substitute materials

### Background

Calcium phosphate (CaP) ceramics have been widely used as bone graft substitute materials owing to their biocompatibility, their chemical similarity to the mineral part of bone and their resorbability in the human body. Clinically used CaPs exist in various crystalline phases such as hydroxyapatite,  $\beta$ - and  $\alpha$ -tricalcium phosphate (TCP), each of which exhibits a very different resorption rate. CaP ceramics are traditionally obtained by high-temperature sintering where the sintering time and temperature determine the resulting crystalline phase. In particular, sintering at below approximately 1120°C results in  $\beta$ -TCP while sintering above this temperature results in  $\alpha$ -TCP. The sintering parameters also affect the final grain size, pore morphology and density.

We have been investigating dense nano-crystalline  $\alpha$ -TCP obtained by slip-casting of a wet-chemically synthesized powder. After sintering at 1200°C, significant amounts of  $\beta$ -TCP were found in these materials, which is in contradiction with thermodynamic theory. Our hypothesis is that this unexpected result is due to a particularly dense nanostructure and a closed porosity in the green state (i.e. before sintering), which induces an isostatic pressure during sintering favoring the crystallographically more compact  $\beta$ -TCP phase. However, the reproducibility of the observed phase transformations is still insufficient.

### Aim

The aim of this Master's thesis project is to investigate the relationships between density, porosity (or pore morphology), specific surface area and the resulting phase compositions.

### Materials and methods

Amorphous calcium phosphate powder will first be synthesized in a precipitation reactor and then thermally transformed to nano-crystalline  $\alpha$ -TCP. Dense bulk samples will then be produced by milling and slip-casting of the powder. Sintering experiments at up to 1200°C will be performed and the phase composition analyzed by x-ray diffraction (XRD). The role of sintering time, temperature as well as milling parameters will be studied. Further characterization will include measurements of the specific surface area (BET) and the plastic limit of the powder (i.e. amount of liquid necessary to obtain a fluid paste).

### Nature of the thesis

Laboratory work: 70%

Data analysis: 30%

### Requirements

Materials science background or similar, interest in materials science and in particular ceramic synthesis

### Supervisors

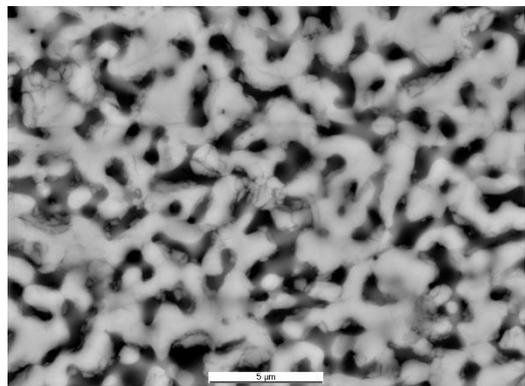
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Pore microstructure of slip-cast  $\alpha$ -TCP samples at early sintering times