Two-step sintering of 3D printed CaP scaffolds for stem cell seeding

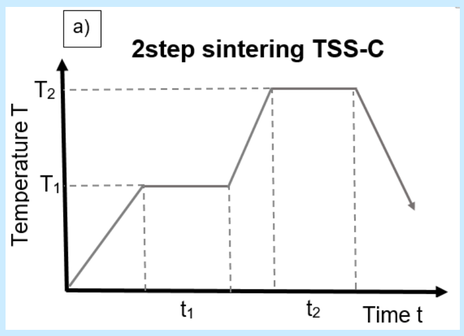
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Motivation

Two-step sintering (TSS) offers several advantages like fine-grained microstructure or controlled porosity, which lead to sophisticated and exact adapted mechanical and functional properties. Typically, TSS methods apply two sintering stages: Step (I) thermal pretreatment and initial heating stage for stimulation of diffusion and grain size re-configuration; Step (II) sintering at elevated temperatures for reaching the final microstructure. The parameters of both stages are essential for the density, remaining pore arrangements and grain size compositions, or in some cases decomposition, amorphisation and recrystallization. Such parameters are temperatures, heating rate, holding times and atmosphere. Particle size distribution and shapes or doping agent influence the successful microstructural design additional. In the present study, a two-step sintering method for 3D printed calcium phosphate powder was explored.

3D printed calcium phosphate bodies should be sintered easily and cost-effective at the costumers laboratory. Pressure less sintering fulfills the demand for minimal price and low technical investment. It may be a future technique for consolidation of costumer printed ceramic pasts.



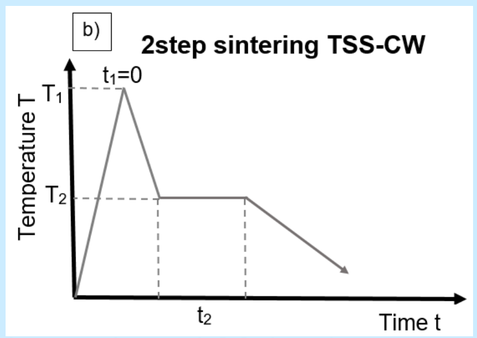


Fig.1: *T-t-regime 2step sintering (TSS) acc. Chu et al. [1] a) and Chen & Wang [2] b)*

2step (TSS-C) sintering produces uniform ceramic bodies in a simple way as suggested by Chu et al. in 1991 (fig. 1a) [1].

Chen and Wang [2] suggested a new methodology of 2step sintering (TSS-CW) in the year 2000 (fig. 1b). The sinter product is rapidly treated at a high temperature and is subsequently cooled and kept at a lower temperature a longer time. Densification occurs with no grain growth in a certain temperature range, which is named by them the “kinetic window”. A prerequisite for full densification is the achievement of a critical density in the first sintering stage, so that there are sufficient triple points throughout the body in stage two.

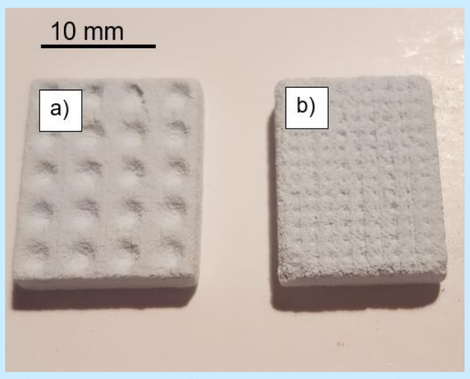


Fig.2: *CaP bodies after 3D printing:*
a) Coarse dimples (∅ = 2mm)
b) Fine dimples (∅ = 0.5 mm)

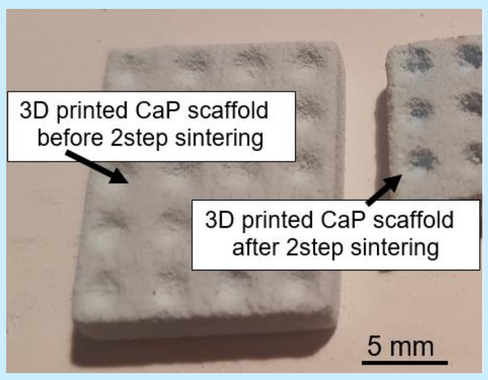
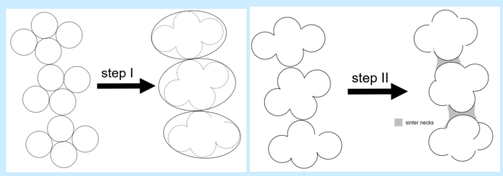


Fig.3: *3D printed CaP bodies before and after 2step sintering*

In the present study, short time 2step sintering (TSS-3Dp) is explored for calcium phosphate CaP ceramic bodies produced by 3D printing (fig.s 2-4) [3]. The motivation of this work is an exploitation of the known advantages of 2step sintering [1,2,4,5].



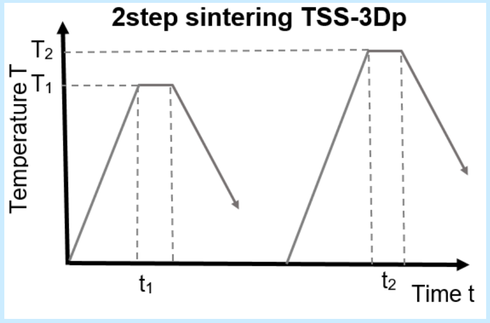


Fig.4: *TSS-3Dp: 2step sintering of 3D printed ceramics*

Additional goals are:

- further reduction of sintering time
- simple and cost effective sintering
- sinter body with open porosity for permeability with liquids (sufficient provision of nutrients for cells & transport away waste from cell metabolism)
- tailoring the porosity, phase composition & strength acc. clinical demands
- infiltration by medical agents (drugs, antiseptic substances etc.)

Results

Scanning electron microscopy (SEM)

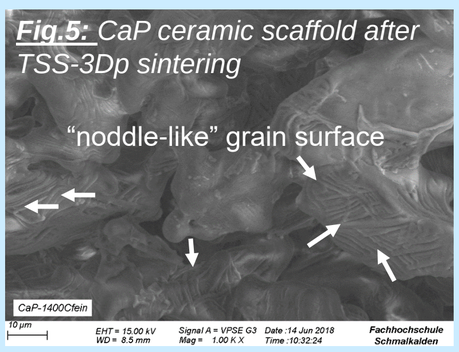


Fig.5: *CaP ceramic scaffold after TSS-3Dp sintering*

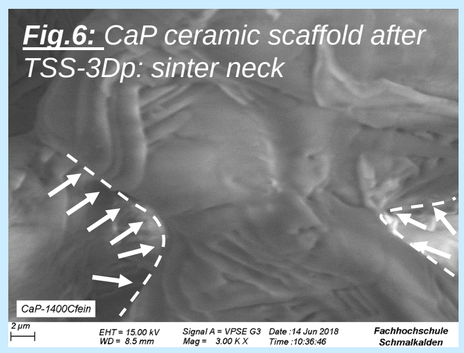


Fig.6: *CaP ceramic scaffold after TSS-3Dp: sinter neck*

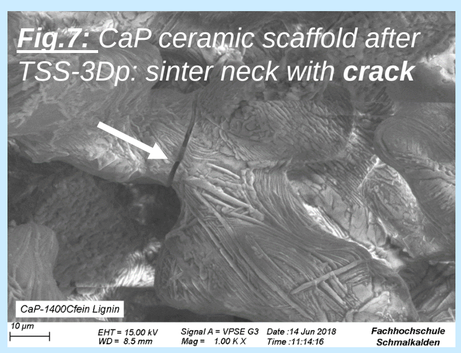


Fig.7: *CaP ceramic scaffold after TSS-3Dp: sinter neck with **crack***

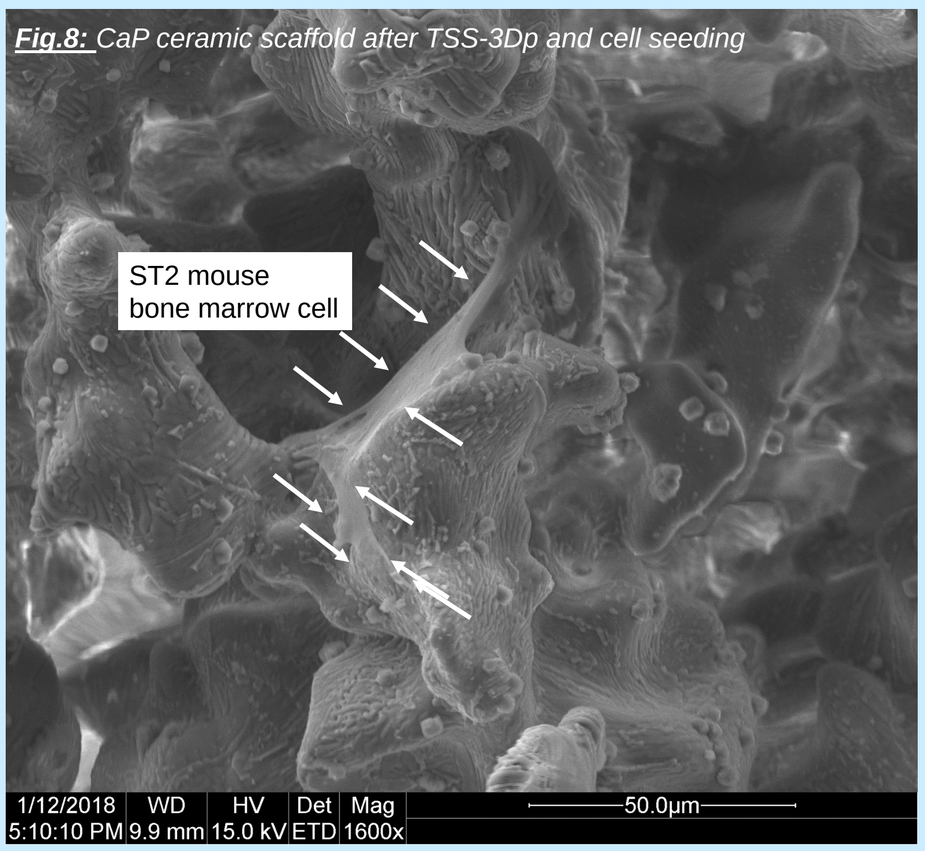


Fig.8: *CaP ceramic scaffold after TSS-3Dp and cell seeding*

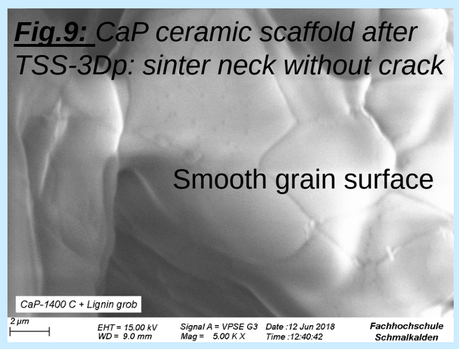


Fig.9: *CaP ceramic scaffold after TSS-3Dp: sinter neck without crack*

SEM was performed after sintering and after cell tests with mouse bone marrow cells ST2.

Surface topography of the bioceramic grains differs heterogeneously.

There are regions of smooth grain surface (fig. 9). The same samples show area of grains with a noddle-like surface topography (fig.s 5-7).

ST2 cells adhere and spread out excellently on the porous CaP scaffolds in any case [3].

Transmission electron microscopy (TEM)

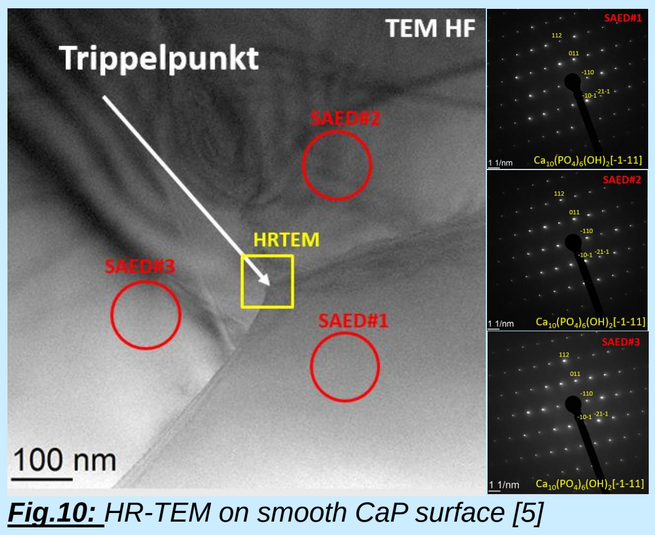


Fig.10: *HR-TEM on smooth CaP surface [5]*

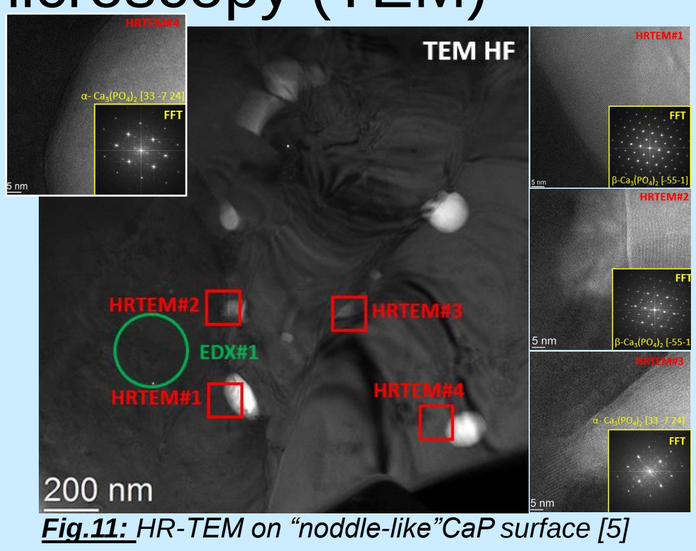


Fig.11: *HR-TEM on “noddle-like”CaP surface [5]*

Xray diffraction

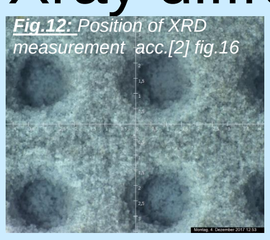


Fig.12: *Position of XRD measurement acc.[2] fig.16*

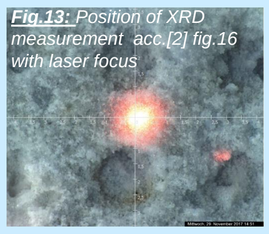


Fig.13: *Position of XRD measurement acc.[2] fig.16 with laser focus*

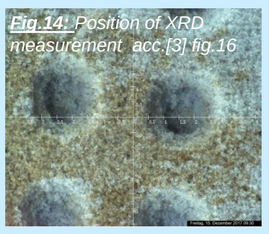


Fig.14: *Position of XRD measurement acc.[3] fig.16*

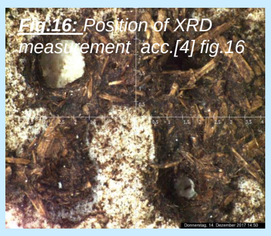


Fig.15: *Position of XRD measurement acc.[4] fig.16*

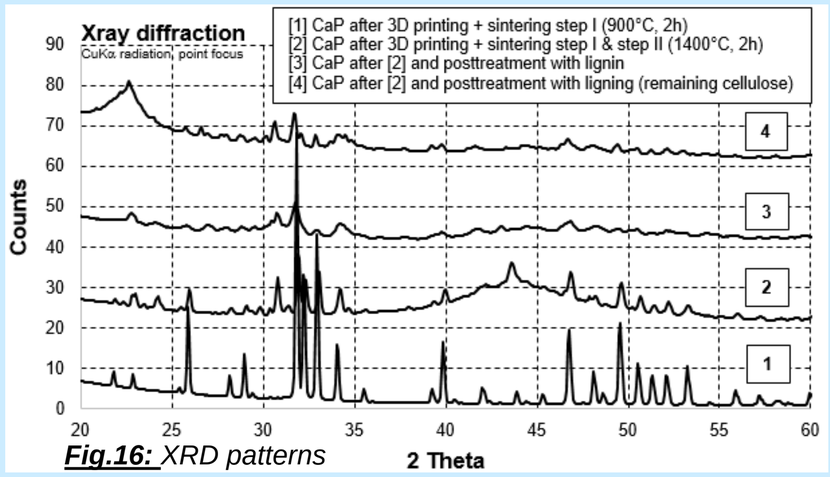


Fig.16: *XRD patterns*

Conclusions

- Scaffolds for stem cell seeding with open porosity and adjusted microstructure need special sintering methodologies.
- 2step sintering of 3D printed CaP scaffolds were explored choosing extreme sintering parameters. Sinter neck formation and heterogeneous phase decomposition is observed.
- Mouse bone marrow cells point to a good bioactivity. Promotion of cell adhesion on the “noddle-like” regions on CaP scaffold surface need to be measured in future work.

References

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