Review Report on PhD Thesis

Faculty: Central European Institute of Technology
Brno University of Technology in Brno

Academic year: 2023/2024

Student: Maliha Siddiqui

Doctoral study program: Advanced Materials and Nanosciences

Field of study: Advanced materials

Supervisor: Doc. Ing. David Salamon, Ph.D.

Reviewer: Ing. Luca Bertolla, Ph.D.

PhD thesis title: The presence of trace additives in advanced ceramics and their influence on sintering

Topicality of doctoral thesis:

The topic of the thesis is of high interest from the point of view of research of materials science, offering some useful notions for advanced ceramics production. The understanding of the role of dopants is indeed a key point to be addressed for the fabrication of both piezo-materials and bone tissue engineering scaffolds.

Meeting the goals set:

The established goals could be summarized at page 33 of the thesis. In my opinion, the candidate has exhaustively fulfilled all the mentioned goals.

Problem solving and dissertation results:

The work is introduced by a theoretical background, where most of the current state of art is summarized. The first part of the work focuses on the doping of β-TCP by $CaCO_3$, with the aim to obtain a biphasic β-TCP/HA. It was found that the extent of transformation can be tuned by the amount of $CaCO_3$ added. Importantly, it has been also found that the incorporation of the dopant did not have any adverse effect on the macrostructure or microstructure of the BCP scaffolds. Another important aspect from the technological standpoint is to ascertain the most suitable consolidation technique of these materials. The candidate delved into three consolidations routes, namely: pressureless, cold-sintering and hydrothermal. The resulting microstructures have been compared, showing significant changes in microstructure. It was shown how doped scaffolds exhibited structural integrity and higher bioactivity, even after hydrothermally treatment.

Concerning the $BaTiO_3$ fabrication, the work explores cold sintering as fast consolidation techniques in order to avoid the impairing of their dielectric performance and Sr-doping as a strategy to effectively lower the Curie temperature (T_c) at room temperature. Also, it was proved that the phase transition from tetragonal to cubic is induced by the simultaneous application of pressure with temperature during cold sintering. The characterization of the newly formed phases was performed by a combination of SEM and TEM microscopy with XRD, Raman and XPS, leading to the conclusion that Sr^{2+} occurs in the form of BST oxide in the BTA matrix. Overall, the candidate provided very interesting results and an exhaustive and well-written thesis, proving her broad knowledge of in the field of dopants for piezo- and bone scaffold materials. Also, the entire work is well embedded into literature.

Importance for practice or development of the discipline:

The results of presented in this thesis represent a contribution in the field of bone tissue scaffolds and piezoceramics technology. For the latter, the main technological hurdle involves difficulties in maintaining the piezoelectric characteristics after thermal treatment. The presented results offer an efficient strategy, namely the Sr^{2+} doping on the piezoelectric performance of $BaTiO_3$, in particular on its influence on key parameters such as T_c and ε_r. These findings may enable a consistent acceleration of the sintering process, by lowering the energy consumption, and thus resulting in more sustainable production.

Concerning TCP samples, it was undisputedly proved that controlled Ca^{2+} doping of TCP samples turned out as a smart method to introduce HA as a secondary phase, with the effect of simultaneously improving the bioactivity and mechanical performance of the scaffolds, two aspects often in competition with each other.

Formal adjustment of the thesis and language level:

The dissertation is well written and organized. The level of results is suitable for publication in international peer-reviewed scientific journals and sufficient to estimate the scientific merits of the PhD applicant. The language used is correct, logical and easy to read.

Questions and comments:

- Beside the dopants used in the present work, which other elements would you suggest as potential candidates for your materials and if any, which are the advantages you forsee?
- Which strategies would you employ, in order to increase the density of the samples?
- In section 4.2.3., page 39, You mentioned annealing routes up to 1100 °C". Can it be still considered an annealing process at such high temperature, or rather an effective sintering step?

Conclusion:

In my opinion, the reviewed thesis fulfils all requirements for obtaining PhD degree. This thesis is ready to be defended orally, in front of respective committee.

In Brno, 20/08/2024

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Ing. Luca Bertolla, Ph.D.