Review Report on PhD Thesis

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

Academic year: **2019/2020**

Student: **Mariano Casas Luna**

Doctoral study program: **Advanced Materials and Nanosciences**

Field of study: **Advanced nanotechnologies and microtechnologies**

Supervisor: **doc. Ing. Ladislav Čelko, Ph.D.**

Reviewer: **doc. RNDr. Karel Obrtlík, CSc.**

PhD thesis title: **Structure and properties of Hydroxyapatite-Magnesium composites produced by the means of current assisted infiltration sintering**

Topicality of doctoral thesis:

The driving force of technical progress is the development of new and more effective materials with improved properties. This is valid also for biomaterials that are employed in components implanted into the human body to replace or augment a natural function of diseased or damaged body parts. Biodegradable implants are a novel class of advanced biomaterials the advantage of which is the elimination of follow-up surgery to remove the implant after the tissue has healed sufficiently. In spite of some unsolved issues, interpenetrated Mg-based alloys and calcium phosphates composites are considered as one of the best alternative of promising biodegradable materials for load-bearing applications with controlled degradation rate.
Thus, it can be concluded that the topic of the doctoral thesis is up-to-date.

Meeting the goals set:

The objectives of the doctoral thesis are stated in section 3. They include the design, manufacture, and characterization of interpenetrated magnesium/calcium-phosphate composites as potential degradable biomaterials for orthopaedics. The doctoral thesis has met the stated objectives completely.

Problem solving and dissertation results:

Calcium phosphates (hydroxyapatite (HA), calcium-deficient HA, and tricalcium phosphate) powders were synthesized and used to produce scaffolds of controlled porosity using the robocasting technique. The scaffolds were infiltrated with pure Mg, Mg+0.2wt. % of Ca and Mg+0.1wt. % of Zn using the current-assisted metal infiltration technique. The Mg alloys were selected based on experimental findings in biocompatibility, corrosion resistance and mechanical properties. Mg alloys and Mg/CaP interpenetrated composites were characterized with micro-computed tomography, optical and scanning electron microscopy, and X-ray diffraction. Besides, uniaxial compression tests and degradation kinetics assessments of the final materials were performed. It was found that different level of decomposition was present in the HA, CDHA, and TCP ceramic scaffolds after infiltration. Comparatively higher stability was observed in the scaffolds made of HA.

Importance for practice or development of the discipline:

The submitted PhD thesis contributes to the development of degradable and bioactive composites for temporary bone fixation implants. Particularly, a combination of additive manufacturing to produce scaffolds of controlled porosity and of a current-assisted metal infiltration technique to manufacture interpenetrated composite structures are of great importance for practice.

Specific routes of Mg/CaP composites production and results of tests and observations to reveal their mechanical, corrosion, and cytotoxicity properties add to the general knowledge of material science.

Formal adjustment of the thesis and language level:

The dissertation 128 pages in length is divided into 7 sections with figures, diagrams and tables included in text and with 168 references. After Introduction, section II shows Literature review with basic characteristics of biomaterials, calcium phosphates (CaP), and Mg and its alloys as degradable biomaterials. Then robocasting of scaffolds, liquid metal infiltration, and Mg/CaP composites are shortly described. Aims of the Thesis are included in section III. Experimental details are described in section IV. Section V presents acquired experimental results and their discussion. Section VI shows main conclusions of the dissertation. Text of the

work is clear and lucid with sporadic typing errors. E.g., on page 85, in the title: "...MG/CAP composites" instead of "...Mg/CaP composites".

Questions and comments:

- Colours of curves in some diagrams (e.g. 21, 22, 23) are not selected properly. Individual curves are difficult to identify.

- Bone tissues of human body are prevailingly strained with variable forces in everyday activity. But the research of mechanical properties is limited mainly to compressive uniaxial tests. Why? Is it difficult to obtain fatigue characteristics of studied materials?

- Fig. 32d shows that the strain of HA preform is double the strain of CDHA preform. Table 11 indicates opposite results. Can you explain? Can you explain stress-strain curve of HA preform?

- What is the grain size of initial pure Mg. Did you observed grain sizes in Mg and its alloys in interpenetrated composites?

- After the current assisted infiltration process the new phases were formed at the ceramic-metal interface. Were they found beneficial or detrimental for potential application in orthopaedics?

Conclusion:

In my opinion, the reviewed thesis fulfils all requirements posed on theses aimed for obtaining PhD degree. The stated objectives were met completely. The PhD student performed complex experiments and observations, obtained original research results, made their analysis and critical evaluation and showed the ability for creative scientific work. Therefore, this thesis is ready to be defended orally, in front of respective committee. After the successful defence I recommend the award of PhD academic degree to Mr. Mariano Casas Luna.

In Brno, 31 August, 2020

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doc. RNDr. Karel Obrtlík, CSc.

CEITEC – Středoevropský technologický institut
Vysoké učení technické v Brně
Purkyňova 123, 612 00 Brno, Česká republika
www.ceitec.cz