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Review Report on PhD Thesis

Faculty/Institute:	Central European Institute of Technology Brno University of Technology in Brno	Academic year: 2021/2022
Student:	Ing. Vladimir Prajzler	
Study program:	Advanced Materials and Nanosciences	
Field of study:	Advanced materials	
Supervisor:	prof. RNDr. Karel Maca, Dr.	
Reviewer:	doc. Ing. Ľuboš Bača, PhD.	

PhD thesis title: Tailoring of microstructure of advanced ceramic materials by conventional and non-conventional sintering approaches.

Topicality of doctoral thesis:

The subject of the doctoral thesis was focused on the research of new sintering procedures for preparation of various ceramics including high purity alumina, 3YSZ, 8YSZ and self-synthesized piezoelectric powders. As the hot topic in the world is reducing energy consumption, shortening of production time and costs, a new procedures and technologies are essential to investigate and compare with the traditional techniques. Present thesis investigated mainly rapid rate sintering (RRS) and flash sintering methods as fast processes for the densification of above-mentioned ceramic materials. Such technologies could improve overall properties of ceramics by affecting their microstructure.

Meeting the goals set:

The goal of the present thesis was to investigate the effect of rapid heating techniques on the microstructure evolution and densification of structural as well as piezoelectric ceramic materials. These goals were clearly and comprehensible formulated. The experimental part is precisely done with huge number of prepared specimens, sintering parameters and techniques used. The analysis and sintering processes were chosen carefully with the focus on the current trends. Their implementation shows high quality of the research performed. The goals of the work were fully achieved.

Problem solving and dissertation results:

The author carefully and comprehensively reviewed the current state of the art with the focus on the sintering (conventional, RRS and FS), grain growth in polycrystalline ceramic materials, introduction, and

assessment of sintering procedures. The attention was also paid to the starting powders as the origin, chemical composition, impurities, particle size and distribution influence their sintering behaviour. The mechanisms of the grain growth during densification by applying of RRS and FS were summarized and compared with the literature.

The present research revealed that the densification of Al_2O_3, 3YSZ and 8 YSZ powders was accelerated using RRS process, although the grain growth was found to be similar as in the case of conventional sintering. FS performed on the 3YSZ powder showed dependence of the microstructure on the current density and other initial parameters. Finally, piezoelectric performance of BCZT, Ce-BCZT and BCST ceramics prepared by different sintering methods was affected by uniformity of microstructure, grain size and impurities.

The results obtained are highly original, and no doubt can be stressed about their novelty. They represent very high quality as well as quantity, presented at conferences and published and/or submitted for publications in top journals in the material science – ceramic filed.

Importance for practice or development of the discipline:

Rapid rate sintering and flash sintering belong to the newest techniques investigated in the last decade as pressureless sintering technologies. Therefore, the comprehensive and detailed investigation of ceramic powders using these techniques could significantly contribute to the technology transfer into the industry. At the same time increasing demands for better and more durable materials can improve the importance of new technologies.

Formal adjustment of the thesis and language level:

The study is rich in introduction and theoretical assessment; it contains an excellent section on the state of the art. Further, the work describes experiments in detail and presents a thorough discussion, where the author is aware of the practical value of his experience. The work cites 116 sources, the author showed his ability to work with literary resources and to build on them. The language is very good; the text is basically free of formal mistakes or typos. Typos and minor issues rarely present in the text or tables (e.g. Table 9 – grain size in nm) do not disturb the reader in any way. However, the description of Figures should contain all necessary information e.g. names of samples although they are mentioned in the text. The graphic design and especially the presented images and schematic illustrations are very useful, clear, and perfectly visualized.

Questions and comments:

1. Did you consider performing the flash sintering study also for piezoelectric materials? If not, why?

2. P. 35 - Did you check possible impurities also in piezo-powders (e.g. due to the milling)?

3. P. 35 – The author mentioned that the annealing at 600 °C was not sufficient for complete Cl removal from YSZ powders. If the annealing at 600 °C didn´t help to remove Cl species from powders (except of 3YB), why did you decide to perform this heating step for all samples? Did you consider sintering of samples without preheating step? Did you analyse Cl content in the

samples pre-sintered at higher temperatures? Could you please suggest the method for the complete elimination of chlorine from powders or compacts?

4. P. 39 - It appears that the relative densities for YSZ discs are highest for 80 °C/min for all samples instead of 100 °C/min. Could you please comment it?

5. P.40 – The author concluded that "The pre-sintering had a negative effect on the microstructural development of the RRS alumina (TM-DAR) sample, where it slightly hindered densification while accelerating the grain growth." Why it is so? Is this feature connected with the initial phase of sintering or bigger initial grains for samples pre-sintered at 1000 °C?

6. P.40 - The author stated that "... a faster heating rate of 1000 °C/min was detrimental to densification of TZ-3Y-E and TZ- 8Y samples as their final densities were lower than after RRS at 100 °C/min while grains were of similar size." Could you explain why?

7. P.41 – Table 14 – under the Table is mentioned "The piezoelectric constant d33 is taken from Ref. [95]." This statement is confusing, indicating that the d33 values were taken from literature and not measured by the author.

8. P.42 - Why does the author select the final sintering temperature of 1525 °C for 3YSZ bars prepared by RRS instead of 1550 °C as in the case of discs?

9. P.76-77 – Random distribution of abnormal grains (AGs) in the microstructure of the TZ 3YB specimens prepared by FS was explained by localization of the electric current, inhomogeneous heat distribution or hot spots. Could be impurities the reason for such effect? Could some impurities be locally segregated due to higher current density on grain boundaries and causes AGG? From Fig.44 is obvious that the AGs were more widely localized in the core of the blackening area and decreases to the surface.

Conclusion:

In my opinion, the dissertation thesis of M.Sc. Vladimir Prajzler fulfils all the requirements of such a work.

I recommend accepting the thesis as a basis for the defence of the dissertation.

In Bratislava, date 29.11.2021

doc. Ing. Ľuboš Bača, PhD.

Reviewer