



Review Report on PhD Thesis

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

Academic year: **2017/2018**

Student: **Vijay Bijalwan**

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

Field of study: **Advanced Materials**

Supervisor: **prof. Timothy William Button, Ph.D.**

Reviewer: **Susana Maria Henriques Olhero, Ph.D.**

PhD thesis title: **Study of the synthesis and processing conditions on the structure and properties of (Ba,Ca)(Ti,Zr)O₃ lead-free ceramics**

Topicality of doctoral thesis: The topic of Vijay's Doctoral Thesis is of current interest worldwide in a scientific point of view, as evidenced from the large number of publications in the area. The topic follows the plan of European Union that intends to restrict the use of hazardous substances such as lead, finding environmentally friendly materials. Even if several lead-free compositions already presented in literature showed stable piezoelectric responses, was still difficult to match the overall performance of PZT. Therefore, continuous and increasing research in this subject presents high impact at both scientific, environmental and technological levels.

So, the novelty as well as scientific level of the thesis is good, considering the importance of the research subject, market requirements and ecological demands.

Meeting the goals set: The main goal of the dissertation is:

- Synthesize lead free BCZT ceramics and optimize the processing conditions to obtain lead free BCZT ceramics sintered at lower temperatures than the usually used in literature that could present similar or even high piezoelectric performance.

For that, the author study the effect of cerium oxide (CeO_2) on the structure and properties of BCZT, by changing several parameters, namely sintering temperature, amounts of Cerium oxide, sintering schedule and Cerium position in perovskite lattice.

According to the results presented and with conclusions obtained it can be confirmed that the formed objectives of the work were positively attained.

Problem solving and dissertation results: Thesis presents several advances in comprehending the structure and properties of BCZT in presence of cerium added in different ways (as additive or being part of crystalline lattice). Highly intensive study in terms of BCZT characterization properties, namely density, microstructure, crystalline structure, dielectric, piezoelectric and ferroelectric properties were presented. Dissertation results are coherent, properly explained and conveniently validated by literature results. In what concerns Cerium effect in BCZT structure and properties, highly advances have been presented in this Thesis and the aims were attained, being possible to say in that sense that “problem was solved”. However, since processing conditions could significantly change the final material properties, several future work could be evaluated in this area, namely to explore colloidal shaping techniques and additive manufacturing to produce BCZT structures for certain applications.

Some results, like addition effects of 1-wt% CeO_2 (section 6(I)) in BCZT properties are out of trend. While author has been advanced some explanations for that, additional techniques, like HRTEM, Rietveld refinement should be used to clarify this result, being this identified as future work in the Thesis.

Importance for practice or development of the discipline: Thesis constitutes a significant contribution to the knowledge and understanding of the field concerned and is a valuable piece of work for students or researchers that would like to continue studying lead free materials. A deeply study of Cerium addition effects in BCZT structure and properties (characterization) have been performed. A proper correlation between all measured properties was evaluated.

Formal adjustment of the thesis and language level: This PhD thesis is well structured and correctly presented. It consists of 5 main chapters, namely Theory and Terminology, Literature Review, Processing and Characterisation (Experimental Procedure), Results and Discussion with 5 separated sections and Conclusions. At the end of dissertation lists of Figure and Tables was presented. List of abbreviations and symbols is presented in the end of the Thesis (Nomenclature). Finally, summary, scientific activity of the PhD student is presented. The thesis is written on 117 pages altogether, and

enriched by figures, equations schemes and tables. The theoretical principles, as well as, the research part were validated with 107 references, where 42 are after 2010.

This thesis is quite well written, although English revision in a number of sentences should be done, as well as, some typing errors should be corrected.

Questions and comments:

- The aims of the thesis should be written in a straight and most appealing way. Reading the objective's as they are, it seems that Vijay's thesis work will not bring any advance in the obtaining of lead free materials. Two objectives presented (last two bullets) are not objectives but work evaluated...
- The novelty of the Thesis work compared to literature should be clearer in dissertation.
- English language and typing errors should be revised. In PhD Thesis document (pdf) I underlined at yellow some sentences or typing errors (spaces, sentences that starts with lower case, long sentences with absence of commas throughout the text, etc...) than in my opinion need revision.
- Pag. 33 – The reason for binder's addition should be written.
- Table 3 format is confusing.
- As presented in a previous work [58], "the particle size of the calcined ceramic powder, and possibly the powder synthesis route, should be carefully selected in order maximize c/a ratios in the sintered ceramics". It should be mentioned in Experimental part of the Thesis the reason to select the milling conditions and calcination procedure of the BCZT powder, or at least add a reference.

Conclusion:

In my opinion, the reviewed thesis fulfill all the requirements posed on theses aimed for obtaining PhD degree and the work developed is worthy of publication in an appropriate high quality scientific journal. This thesis is ready to be defended orally, in front of respective committee.

Some suggestions given in "questions and comments" section are not mandatory, but can be accommodated in a possible revised version.

In Aveiro, 28 September 2018


Susana Maria Henriques Olhero, Ph.D.