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

Student: Hua Tan

Doctoral study program: Advanced Materials and Nanosciences
Field of study: Advanced materials

Supervisor: Assoc. prof. David Salamon, Ph.D.
Reviewer: Ing. Jaroslav Sedláček, PhD.

PhD thesis title: Extremely fast sintering of advanced ceramic materials

Topicality of doctoral thesis:

Despite the fact that the spark plasma sintering technique (SPS) is known for decades and thus is not so novel as also author mentioned in Introduction, there is still lack of information on the processes which occur during sintering of mainly non-conductive ceramics. The industry is looking for low cost technologies for producing the materials, actually also such a technologies that are environmentally friendly. Some results presented in this work clearly shown that SPS could met these criteria, moreover, materials with added value can be prepared. The topic of proposed PhD thesis is therefore up to date.

Meeting the goals set:

Impact of SPS processing parameters on the sintering mechanisms of ceramics has been studied in proposed work. Overall, it can be considered that all the goals has been met, however, some of results or findings need to be completed by further analysis, in this I strongly agree with author.

Problem solving and dissertation results:

PhD student demonstrated the multidisciplinarity of thinking mainly by involving the theoretical approach (modeling) in discussing of obtained results and thus the effort of complex description of problems. This I rate it very positively. However, some of the presented results are not clearly discussed due to missed analyses (chemical, phase, microstructural, etc.) either as a consequence of lack of a time or other reasons unkown to me. Despite this, reviewed thesis demonstrate the independent thinking of author and possible room for improvement of his abilities.

Importance for practice or development of the discipline:
Parts of reviewed thesis related to the impact of pulse pattern and timing of applied pressure are very interesting results, not published yet (authors manuscript is under review) and represents the widening of basic knowledge on SPS as well as other field assisted sintering techniques. However, it is necessary to answer some open questions, mentioned also in reviewed thesis.

**Formal adjustment of the thesis and language level:**

Formally there is almost nothing to recommend for adjustment from the point of structure and language. Thesis is structured logically, sometimes written in poetic English without any disturbing mistakes. However, some mistakes were found in figures, p. 39 – Fig. 5-7 missing microstructure (or wrong figure numbering in text below?), p. 81 – wrong x-axis labeling of Fig. 5-34.

(4) The study is duly completed by a state doctoral examination and the defense of a dissertation, which proves the ability and readiness for independent activity in research or development or for independent theoretical and creative artistic activity. The dissertation must include original and published results or results accepted for publication.)

**Questions and comments:**

1) There is a lack of information on chemical composition of starting powder, mainly metals used for alumina composites. Could you please provide these within your presentation? As example, you used iron powder with 90% purity level, i.e. 10% is unknown phase that can affect the sintering of alumina or other „surprising” findings.

2) Did you analysed the starting powders or mixtures, form the point of phases presented in starting powders or formed during mixing? You have mentioned the presence of iron may affect the phase transformation of alumina, however, you did not checked it.

3) Similarly to previous, why you did not analysed alumina samples after sintering as was done in case of boron carbide samples?

4) There is a description of SPS experiment on page 26. Could you please explain me the reason of pre-heating described in the last section and followed on next page. Is it something like cleaning process?

5) According to data provided in Experimental, size of iron powder was below 100 microns. Does it mean that the ball milling or SPS was so effective in terms of refinement of iron particles, see microstructure on Fig. 5-4. It seems that average iron particle size is approx. 20 μm. Moreover, iron particle (Fig. 5-4c) seems to have reaction zone (different material contrast either by different phase present or low quality of micrograph). Could you please explain this, as you mentioned no reaction between iron and alumina, however without any relevant analysis.

6) Part related to the pulse pattern effect is very well discussed and I agree with you that there are open questions need to be studied in detail. However, you mentioned the influence of type of DC generator. Could you please explain this more detailly? I suppose that construction of DC
generator can only influence the stability of pulses (you observed that despite the set up to strict value of pulse in fact there was a range or deviation). Is the needle shaking so important parameter? May it be explained by insufficient damping effect of pulses of volt- and amperemeter?

7) Are the findings of Grasso’s research valid for non-conductive alumina? In cited work (52) they used a system of graphite die with graphite sample, i.e. materials with same properties. In case of alumina, the current goes through the graphite die. Pressure effect was attributed to the better contact between punches and die, but with conductive sample. Did you observed something similar for boron carbide (effect of pressure)?

8) Sintering of boron carbide doped with Ti result in formation of „textured“ ellipsoidal agglomerates. Could you please provide the information of direction of pressing force? Do you expect that the mixing level was sufficient, in terms of homogeneity? Did you observed some excess of carbon in your samples after SPS?

Conclusion:

Above mentioned questions come from the lack of data in proposed thesis and could be valuable for PhD student (author) in explaining some questions and thus to increase the quality of thesis. However, despite some missing information and thus vague interpretation of results I suppose that reviewed PhD thesis is valuable. Mainly the part related to pulse pattern effect is very interesting and valuable for research as well as industrial applications of SPS technique.

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

In Bratislava, date 10.5.2020

Ing. Jaroslav Sedláček, PhD.