Oponentní posudek disertační práce

Ústav: Středoevropský technologický institut VUT Akademický rok: **2023/2024**

Student (ka): **Ing. Vojtěch Mařák**

Doktorský studijní program: **Pokročilé materiály a nanovědy**

Studijní odbor: **Pokročilé materiály**

Vedoucí disertační práce: **Ing. Daniel Drdlík, Ph.D.**

Oponent disertační práce**: Ing. Monika Michálková, Ph.D.**

Název disertační práce: Keramické materiály a kompozity pro pokročilé aplikace

Aktuálnost tématu disertační práce:
The author has presented a systematic topical work on advanced oxide ceramics and laminate composites. The indisputable topicality is underlined especially after implementing the achieved knowledge in tailoring individual processing steps and employing novel fabrication and treatment methods to optimise the desired performance of the materials.

Splnění stanovených cílů:
The thesis set out 6 core objectives, and all were met.

Postup řešení problému a výsledky disertace:
The author systematically and conscientiously addressed the challenges, considering all relevant experimental parameters that influence the properties of selected oxide ceramics. Additionally, the comprehensive exploration of factors impacting the properties of the materials, along with the thorough characterization of the materials, is commendable.

Význam pro praxi nebo rozvoj vědního oboru:
The published results significantly contribute to the field of oxide ceramics preparation. The elaboration of the chosen topic provides a comprehensive and up-to-date view of the problems of the processing and sintering of oxide ceramics. The findings published and submitted in scientific journals (6+2) hold substantial importance for both the industrial and academic sectors, given their comprehensive nature and in-depth analysis. The author explored the plasma treatment of hydroxyapatite powder, which enabled electrophoretic deposition without the use of toxic stabilizers, resulting in the production of denser and crack-free coatings. Additionally, through optimized co-precipitation, thermomiotic aluminum tungstate, recognized for its near-zero coefficient of thermal expansion, was synthesized to yield a powder with enhanced sintering properties. The material was then densified using rapid pressure-less sintering and spark plasma sintering, achieving the highest density at the lowest temperature to date.

Formální úprava disertační práce a její jazyková úroveň:
The thesis meets the criteria set for this type of work linguistically and formally. It is 87 pages divided into 10 chapters, and formal errors occur only sporadically.

Zda dizertační práce splňuje podmínky uvedené v § 47 odst. 4 zákona:
(4) Studium se řádně ukončuje státní doktorskou zkouškou a obhajobou disertační práce, kterými se prokazuje schopnost a připravenost k samostatné činnosti v oblasti výzkumu nebo vývoje nebo k samostatné teoretické a tvůrčí umělecké činnosti. Disertační práce musí obsahovat původní a uveřejněné výsledky nebo výsledky přijaté k uveřejnění.[1]).
Práca spĺňa podmienky uvedené v zákone § 47 odst. 4.

Pripomienky

Page 30—sentence: “A total weight loss of 18.7% was recorded, but the weight loss above 570 °C was negligible, suggesting that structural changes were complete.“Although the author later rightly writes about crystallization at 635°C from the DSC curve, the last part of the cited sentence is incorrect because structural changes can not be predicted according to the TG curve.

Fig. 22 is a bit harder to understand. The overlapping peaks at positions close to 32 in the case of zirconia doping (e) seem to be wrongly signed.

Tab. 7 - K_{IC} of $BaTiAl_6O_{12}$ compound would be useful here.

Fig. 32 - very hard to understand due to the lack of explanation in the fig - e.g. T and composition, type of sintering...

[1] *§ 10 zákona č. 35/1965 Sb., o dílech literárních, vědeckých a uměleckých (autorský zákon).*

Otázky

1. During the preparation of $Al_2W_3O_{12}$ thermomiotics, $Na_2WO_4 \cdot 2H_2O$ is used. Have you observed any contamination of your samples by sodium? What would be the influence of sodium on sintering/microstructure?
2. Page 28 – while inserting the stainless steel orthopaedic screw into the bone, is the coating able to sustain?
3. **Fig. 8, Fig 9, Fig 13a**: these figures are connected a lot.
   In **Fig. 8**, the crystalization of $Al_2W_3O_{12}$ powder is observed at 635°C. However, amorphous materials have a glass transition temperature interval, for $Al_2W_3O_{12}$ powder seems to be around 590°C. This is not mentioned in the work, but it is an important property of an amorphous material. In this temperature interval, the sample starts to flow in a glass transition interval.
   From **Figure 9**, we can observe that the amorphous hump is still present in the sample after being calcinated at 620°C. So, the powder is not fully crystalline and contains an amorphous phase.
   Fig. 13a shows that the first phase of shrinkage during sintering was observed at a temperature of about 590 °C, which, according to the DSC record, would be consistent with the glass transition temperature. Could you please verify and comment on these statements? Moreover, since no weight loss is observed above 500 °C and the colouration is caused by oxygen vacancies, would it not be worthwhile to sinter a powder with a higher proportion of the amorphous phase?
4. **Fig. 26**—Why does the singing curve rise twice due to volume phase change? If so, wouldn't it be better to stop the experiment at the point of greatest shrinkage to achieve maximum density?
5. **Fig. 27** - The ratio among the intensities of samples sintered at 1450 °C SPS and 1450°C has changed - really only $BaTiAl_6O_{12}$ was present at 1450°C? Additionally, in Fig. 28, a secondary phase was detected; could that be connected with the change of ratio among intensities in XRD?

Celkové zhodnocení disertační práce:
The author has demonstrated strong creative skills in the chosen area of research and has thoroughly engaged with the subject. I recommend defending the submitted thesis for the award of the academic degree philosophy doctor "PhD."

Disertační práci Ing. Vojtěcha Mařáka **doporučuji** k obhajobě pro udělení akademického titulu “doktor“ (Ph.D.).

V…Trenčíne……dne 5.6.2024 ……

Ing. Monika Michálková, Ph.D.

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