Oponent's review of PhD dissertation of Richa Saggar

Preparation and properties of the glass matrix composites reinforced by 1D and 2D boron nitride nanoparticles

Dissertation of Richa Saggar deals with composites based on borosilicate glass (BS) and boron nitride (BN) nanoparticles with the aim to increase the fracture toughness and strength of borosilicate glass by adding BN nanoparticles. The specific objectives included characterization of commercial nanotubes of boron nitride (BNNT) and the use them for preparation of borosilicate glass-BN (BS-BNNT) composites, preparation of BN nanosheets (BNNS) by exfoliation in the liquid phase and the use it for manufacture of borosilicate glass-BN (BS-BNNS) composites. Ceramographic analysis of composites and study of their mechanical properties and examining the fracture micromechanics and fracture micromechanisms of borosilicate glass-BN composites were final tasks of dissertation.
Dissertation has a somewhat unusual structure of chapters. It contains standard introduction, literature review and objectives. Then follow two separate chapters, the first of which relates to borosilicate glass reinforced by commercial BN nanotubes (BNNT), the second focuses on the synthesis of BN nanosheets (BNNS) and preparation of borosilicate glass reinforced by BNNS. Each of these chapters contains a separate experimental part (but without any designation), results and discussion. The results of both chapters are compared in separate chapter entitled "Comparative Analysis". Conclusion and references are listed at the end of the dissertation. The unusual structure of the dissertation requires great attention when reading.
The theoretical part is handled well and therefore I have no fundamental objections; the text and tables are clear and graphics are adequately presented. However in Sec. 2.2.2 are given inaccuracies regarding stabilization mechanisms of colloidal dispersion, and in Fig. 2.8 is not shown electrostatic stabilization but electrosteric one. The aims of the work are described clearly and the results of PhD candidate match the goals. The achieved results and their adequate discussion are presented in three chapters, "Boron Nitride Nanotubes Reinforced Composites Borosilicate" and "Synthesis of Boron Nitride and Boron Nitride Nanosheets Nanosheets Borosilicate Reinforced Composites" and in comparative chapter "Comparative analysis".

The thesis contains some inaccuracies or unclear wording, which must be explained by PhD candidate at thesis defence:

Sec. 4.4.1, 4.5.1, 4.6.1, 5.4.4: Measurement of zeta potential was carried out in ethanolic dispersions of BN and BS. The values of the zeta potentials were negative (-17.9 and -21.3 mV) at pH around 7.0. After washing the BS and BN in HCl and HNO3 the zeta potential increased to -0.578 mV, pH dropped to 4.18. The results are not explained adequately and properly.
Question1: Which procedure was used for pH measurements of ethanolic BS and BN dispersions? Can be used for pH measurement of ethanolic, non-aqueous a pH meter calibrated by aqueous standards? How do you explain the negative zeta potential of the dispersions in "acidic" environment?
Question2: What is the meaning of sentence "Flow of liquid around the solid surface renders charging the surface which is neutralized by the attraction of opposite charge present in the solvent forming an electric double layer (EDL). Zeta potential helps to determine the strength and polarity of the EDL. "

Sec. 4.5.1, 5.5.1: Thermogravimetric analysis of BN showed oxidation of BN at a temperature

above 600° C.
Question3: What is the mechanism of BN oxidation in air and what products can arise? Can oxidation of the BS-BNNT composites take place during sintering at the temperature 900° C?

Sec. 4.6.1 bubbles in BS-BNNT composites
Question4: Which Fe impurities can evaporate and bubbling at 900 ° C (in the course of SPS sintering)? May be "bubbles" formed due to incomplete removal of pores during sintering?

Sec. 5.1.2 Preparation BN nanosheets
Question5: Exfoliation BN was performed by ultrasound treatment for 24 hours. Sonotrode made of Ti immersed in BN dispersion is subjected to intense cavitation abrasion. Was the content of Ti in exfoliated BN evaluated?

Despite of shortcomings Richa Saggar submitted a dissertation that meet the objectives of the work and bring these new, positive developments in the field of glass composite materials reinforced with nanoparticles (nanotubes, nanosheets) of boron nitride:
Fracture toughness showed an increase of ~30% for 5 wt% BNNT reinforcement compared to pure glass.
Improvement in the fracture toughness by ~45% each for 5 wt% of BNNS reinforced BS composite (1.10 $MPa.m^{1/2}$) was observed in comparison to pure glass (0.76 $MPa.m^{1/2}$).
Improvement in the flexural strength by ~45% each for 5 wt% of BNNS reinforced BS composite (~119 MPa) was observed in comparison to pure glass (~82 MPa).
A reduction of ~23% of coefficient of friction was observed by incorporating 5 wt% of BNNS.

Conclusion:

PhD candidate has shown that it is capable of independent, creative work in the field of material science. I therefore recommend that the dissertation, despite the shortcomings identified in the report, would be submitted to the defence and PhD degree awarded after succesfull defence.

Brno, December 5, 2016

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