CZECH TECHNICAL UNIVERSITY IN PRAGUE
Faculty of Civil Engineering
Department of Science and Research
Thákurova 7, 166 29 Praha 6

e–mail: obhajoby@fsv.cvut.cz tel.: 2 2435 8736

Opponent's review of the Doctoral Thesis

Candidate Ing. Jiří Němeček

Title of the doctoral thesis Micro-Scale Fracture Properties of Cementitious Composites

Study Programme Physical and Material Engineering

Tutor doc. Ing. Jiří Němeček, Ph.D., DSc.
prof. Ing. Petr Štemberk, Ph.D., D.Eng.

Opponent Doc. Ing. Vít Šmilauer, Ph.D., DSc.
e-mail vit.smilauer@fsv.cvut.cz

Topicality of the doctoral thesis theme

Commentary: Highly interesting topic relying on novel experimental techniques (high-speed nanoindentation with modulus mapping & peak force quantitative nanomechanial mapping, focused ion beam milling, scratch tests with acoustic emission, push to pull device). The thesis uses those techniques for Portland cement pastes, alkali-activated materials and blended systems over hydration periods to access local material properties. Micro-scale technique provides interesting insight on ~20 um long samples in direct tension.

☒ excellent	☐ above average	☐ average	☐ below average	☐ poor

Fulfilment of the doctoral thesis objectives

Commentary: The thesis fulfilled all mentioned research objectives; characterization of elastic/fracture properties of binders down to individual phases, damage quantification during SEM / FIB exposures, scratch testing and detection of weakest links in a composite.

☒ excellent	☐ above average	☐ average	☐ below average	☐ poor

Research methods and procedures

Commentary: Novel experimental methods were used in the thesis, particularly devices for accelerated mechanical property mapping (XPM) by Bruker-Hysitron, speeding up loading an order of magnitude. Other methods cover atomic force microscope, nanoindentation, FIB etc. Experimental methods lead to identification of elastic moduli, viscoelastic behavior, tensile strength and fracture energy above all. Fabrication of micro-beams belongs to significant contributions of the thesis, together with damage quantification during the milling. Differences in experimental results were partially explained on some cases, more discussion is welcome to shed light on underlying mechanisms or linearity with respect to loading.

☒ excellent	☐ above average	☐ average	☐ below average	☐ poor

Results of the doctoral thesis – dissertant's concrete achievements

Commentary: Individual tests show excellent reproducibility on cementitious systems and consistency with published data, if available. Mutual comparisons reveal several phenomena, such as strain rate effects or the effect of holding phase. The interpretation used FE analysis

with strain dependent viscoplasticity. The critical issue is permanent (fracture-plastic) deformation induced by high stress under the tip, which distorts linearity and interpretation based often on elastic half-space solution. The thesis brings unique results never published before, such as influence of vacuum (drying) on C-S-H-dominant fracture properties. Experimental methods of cantilever beams explored different milling energies, demonstrating material damage due to high temperatures. Softening part was achieved on notched cantilever beams, allowing to determine fracture energy quite accurately.

☒ excellent	☐ above average	☐ average	☐ below average	☐ poor

Importance for practice and for development within a branch of science

Commentary: The thesis demonstrates various techniques for mechanical assessment of local properties. This opens wide possibility for heterogeneous material analysis on pure and blended cementitious systems. The results nicely illustrate elastic moduli compatibility between outer C-S-H and N-A-S-H gel, paving a way to binary/ternary alkali-activated blended systems. The results show that macroscopic tensile properties are orders of magnitude lower than micromechanical tensile properties, showing strength scaling across scales and revealing other mechanisms contributing to reduction such as microcracks. Very unique experiments were carried out on C-S-H gel cantilever beams, demonstrating that vacuum leads to an of increase 2.3-2.5 times of the tensile strength and increase of elastic moduli, contradicting previous indirect experiments by Sereda [196] contaminated apparently by microcracking. Such understanding can lead to new generation of materials utilizing better their potential. The results improve multiscale models with strength scaling, validating directly more scales.

☒ excellent	☐ above average	☐ average	☐ below average	☐ poor

Formal layout of the doctoral thesis and the level of language used

Commentary: The layout is well arranged. The thesis documents nicely the result on several illustrative images, such as Figs. 2.4-2.7 for phase segmentation or Fig. 6.12 for histograms. Only minor mistakes were found, such as labels in Figure 2.1, Power/Powers, molar balance in Eq. (2.8) etc.

☐ excellent	☒ above average	☐ average	☐ below average	☐ poor

Remarks

1. What is linearity of dynamic mechanical analysis (nanoDMA) with regards to stress concentration under the tip (storage and loss modulus)? Results between micro-bending tests and microindentation show large difference in recovery (31-41% vs. above 80%) [Y. Gan et al., CCR, 2021]?
2. Portlandite is known to be highly anisotropic. What is the impact of crystal orientation on fracture toughness?
3. Apparent Young's moduli differ in nanoindentation and XPM by 10%, which is explained by viscoplastic rate effect. Similar rate effect exists in viscoelasticity by considering material ageing even for very short loading times. Did you try to elaborate this idea of solidification theory used for example in creep model B3?

Final assessment of the doctoral thesis

The thesis describes novel experimental approaches with unique results. Results were published on several conferences and high-impact journals by a Ph.D. candidate, demonstrating his ability to conduct research and interpret the results.

Following a successful defence of the doctoral thesis I recommend the granting of the Ph.D. degree		
	yes ☒	***no*** ☐

Date: Oct 5, 2021

Opponent's signature:.....