Review Report on PhD Thesis

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

Academic year: **2021/2022**

Student: **Pavel Komarov**

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

Field of study: **Advanced nanotechnologies and microtechnologies**

Supervisor: **doc. Ing. Ladislav Čelko, Ph.D.**

Reviewer**:** **doc. Ing. Pavel Ctibor, Ph.D.**

PhD thesis title**: Advanced hydrophobic and hydrophilic surface treatments for non-nuclear energetics**

Topicality of doctoral thesis: The doctoral has two focuses
- the development of a technological procedure to fabricate hydrophilic/hydrophobic coatings from wear-resistant materials utilizing thermal spraying technology;
- a detailed investigation of deposited coatings, analysis of their mechanical properties and the robustness of their wetting behavior.

The purpose of this initiative is to replace in future the existing production ways of superhydrophilic and superhydrophobic surfaces with more efficient processes based on thermal spraying, which is potentially capable also to provide sufficient robustness of the hydrophilic/hydrophobic wetting behavior, not easily accessible for the existing approaches.

Meeting the goals set: Several wear-resistant ceramic and cermet coatings, namely ZrO_2-Y_2O_3, Al_2O_3, Cr_2O_3-SiO_2-TiO_2 and WC-Co-Cr, were produced by means of atmospheric plasma spraying. The topography and the wetting behavior of these experimental coatings were studied in the i) as-sprayed, ii) ground and iii) polished states. All the coatings showed **hydrophilic behavior** with the *Wenzel wetting regime* due to the presence of pores/cracks present in the coatings, which resulted in a gradual infiltration of the water droplets into the coatings.

- In the more detailed study on variously sprayed YSZ coatings the previous character was confirmed with two exceptions – i.e. the denser flat columnar microstructure of the A3-D YSZ

coating (i.e. Axial torch, fast cooling) prevented the penetration of water into the coating, resulting in **hydrophobic** behavior with the *Cassie-Baxter wetting state*;
and the rough surface of the columns, as well as the high surface area, of the WSP-H YSZ sample provided **superhydrophobic** behavior with the *Cassie-Baxter wetting state*.

- The additional **RF-plasma** jet surface treatment led to the surface modification represented by an organosilicon layer with dendritic structure composed of nano-sized particles. Such a layer provided transition of wetting behavior from hydrophilic and hydrophobic states to **superhydrophobic** one with the *Cassie-Baxter wetting regime*.

Another coating technique, i.e. high-velocity oxy-fuel was applied for spraying of WC-Co-Cr cermet materials. A simple way to improve surface hydrophobicity based on a **Si-oil treatment** was provided. All cermet coatings were hydrophilic in the polished state, but hydrophobic and almost superhydrophobic in the as-sprayed state. The nano-scale topographical features of the surfaces caused (in a particular case) that only 3.8 % of the water droplet surface was in contact with the coating's surface.

Problem solving and dissertation results:

Various coating techniques and various parameters set-ups were employed. Various feedstock powder sizes and in case of the cermet also various carbide particle sizes were used.
Surface characterizations via XRF, XPS, SEM and XRD were done. Surface roughness was evaluate also by means of enhanced, not very often reported, parameters like kurtosis and skewness.
The robustness of hydrophobic/superhydrophobic behavior were evaluated by microhardness measurements, cavitation erosion, slurry abrasion response and unconventional non-destructive tests. The sessile droplet method was tested.
The slurry abrasion response test and the cavitation erosion using a ultrasound apparatus were applied for testing the wear resistance not of the coating itself but the wetting surfaces. Some modifications of the most common approach to these tests were done. The "acid-phobic" performance was also evaluated. Besides the mostly expected sessile droplet test also the self-cleaning test using tilted samples and muddy-water transportation test were applied for evaluating the wetting behavior.

Importance for practice or development of the discipline:

The endurance tests of the wetting character of the original surface subjected to wear showed that the hydrophobicity of the WC-Co-Cr cermet coatings was robust enough even for demanding sacrificial industrial applications. Interestingly, it was observed that the produced wear tracks played an amplification role in hydrophobicity due to an enlarged surface area. Silicone oil

surface treatment was able to improve also the corrosion resistance and increase the service lifetime of the WC-Co-Cr coatings.

Formal adjustment of the thesis and language level:

The thesis is sure without serious formal mistakes – the general look is very good, use of citations is correct. Language errors are nearly absenting.

The student is also author of various papers in the review journal, in at least of three cases is Pavel Komarov the first author.

Questions and comments: In the Fig. 16 and in the paragraph just below them are really misleading statements that WC-Co-Cr coating were made also by APS instead of HVOF.

In Tab. 5 the column setting is unclear – the important info: which powder size and carbide size belong to which coating designation, is provided as non-reader-friendly.

In Chapter 5.1.1 the term "segmentation cracks" is absenting, which decreases the clarity of the interpretation.

In Tab. 6 there is material Cr2O3-SiO2-TiO2 but in the phase analysis column there is any TiO2, any Ti – where are phases associated with this component? Evaporated (?) – according my experience not. Overlapping of peaks (?) – which and why?

On page 61 the is "low density of YSZ (~ 2 g/ cm3)". I think the density of YSZ is not so low.

Conclusion:

The work is well treated, the design of experiments deliberately arranged, the measurements correctly performed, and the interpretation of all result thoroughly done.

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

In Prague, date 21.2.2022

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doc. Ing. Pavel Ctibor, Ph.D.

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