Review on the Dissertation thesis of Ing. Hadi Husain

Goals, concepts and methods:
The goal of the study was collecting free particles, worn-out at tribology tests, and prepare a risk assessment. The estimation of risk factor for plasma-welded material on tribologically treated surfaces was shown by possible impurities, generated during this process, on the environment and possible proof improvement for wear resistance. The risk assessment was prepared based on rules of the Agency of European Union regulations and Standards.

The dissertation is focused on cermets as promising materials to be used in tribological applications. The aim was to summarize and estimate risk factors, caused by combining Nickel alloy 625 and Boron Carbide particles in plasma powder transferred arc welding (PPTAW) technique. Boron Carbide (B_4C) ceramic was selected because of its high temperature resistance and considerable hardness and Inconel® metal (i.e. the Ni alloy 625) was selected as a corrosion resistant material with high tensile strength, well preserved to elevated temperatures, excellent weld ability and creep characteristics. The final composition of the cermet was Ni 625: 70% + cubic phase B_4C: 30%.
However, nickel is one of the most common allergens worldwide, causing allergic contact dermatitis. Nickel particles could have great negative impact on working atmosphere in tribological areas.
Cobalt, which content in the Inconel® material is only about 1 percent, is well known with its carcinogenic effect, and due to this was it also included in the scope of the dissertation. Boron carbide was considered as the less dangerous from the addressed components.
The organization of the thesis is as follows: After the Introduction, Chapter 2 reviews the necessary background and Chapter 3 presents the existing works on the addressed materials and the principles of PPTAW technique. Chapter 4 summarizes endotoxin tests for micromaterials and nanomaterials in tribology. Chapter 5, presents the practical experiments and their results. Chapter 6 discusses the results and Chapter 7 concludes what has been done in this work and what are possible future steps.

Various endotoxin presence in-vitro tests like the LAL test CSN EN ISO 29701 (Limulus amebocyte lysate) are mentioned, but the merit of the work is fully in an inorganic

matter - in the field of materials engineering, tribology and plasma-assisted processing of cermets.

Heat Treatment before the tribology tests was necessary for the precipitation of the strengthening phases and improvement of the mechanical properties (based on the literature). The configuration *Ball on flat disk* was used for the tribology test. The released particles were characterized via confocal microscopy. Their morphology was observed in details by SEM and chemical composition investigated by EDS.

Main findings:
It was shown that Ni and Co are both present in submicrometric particles released during tribological tests. Samples with heat treatment had higher average material loss during the tribology test. Always, when both – ball and flat tablet – were heat treated, the quantity of released Ni and Co particles was higher. This probably mentioned that treated samples were harder and more brittle. The wear resistance of the heat-treated samples was partly improved (lower mass loss). - The goals of the thesis were reached.
Weak points of the work (and possible questions):
A Figure of PPTAW arrangement is mentioned in the text (page 10), but in fact is it missing.

Tables in the Sub-chapter 4.9: The data are displayed with a precision to 0.1 nm. Is it a realistic precision of the used confocal microscope? The comments on this topic are missing.

Carbon was seen as well by EDS in the finest particles. It probably comes from B_4C. However, the comment of carbon role from the health risk viewpoint is missing.
Speculation of the reviewer: Heat treatment improves wear resistance, but as a result, more and finer particles of Ni and Co are released at the test (instead of less and bigger particles from the as-deposited material) - is it like this? Also, the thermal treatment is good for the machine service economy but bad for the safety of the staff? Is interpretation like this plausible or not?

Relevance for the industry:
The relevance for the industrial practice lies mainly in demonstration of the harmful particles released by tribo-action on the addressed materials. Persons handling with machine parts in wear action are exposed to dangerous materials (Ni and Co

in this case). Maximum safety precautions are necessary. - This study opens a broad topic, which would need employment of a really interdisciplinary team, including medical doctors, to perform in-vitro or in-vivo tests in future.

Summary:
The obtained results represent good collection of data, whose more comprehensive discussion and more innovative interpretation would bring, probably, even more knowledge. The dissertation is rather conservative, very vigilant to say some may be contradictory statements - that is why its final value is only average, not a really excellent.
However, it fulfilled the necessary criteria for PhD thesis and I can recommend its defense.

In Prague, 19th of August
Assoc. prof. Pavel CTIBOR, Ph.D.