Opponent´s opinion

of the Dissertation Thesis

Effect of Microstructure on Fatigue of Superelastic NiTi Wires

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Degree Programme: Application of Natural Sciences

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Unique superelastic and shape memory properties of NiTi alloys have been utilized in many engineering applications, especially in medicine and automotive industry. However, some applications are severely limited by the fatigue life of components made of NiTi alloys. In the superelastic regime in tension the fatigue life is bounded to thousands of cycles. Better understanding of microstructure – fatigue relationships in NiTi alloys requires more experimental data. The main goal of the dissertation was to define the effects of microstructure, stress and temperature on the functional degradation and fatigue life of NiTi wires in the superelastic regime. The topic of the dissertation can be classified as very useful. The experimental activities were carried out in a close collaboration with the Institute of Physics of the Czech Academy of Sciences, Department of Functional Materials (supervisor specialist: RNDr. Petr Šittner, CSc.), which has a very good position in the research of shape memory alloys worldwide.

The work includes a very complex set of results dealing with thermomechanical tensile tests and fatigue tests on NiTi wires in a wide temperature interval. These results are supplemented by very sophisticated structural investigations. Microstructure characterization was carried out using a number of modern techniques: SEM, TEM and SANS. Applications of the site specific method of thin foils preparation (FIB) made it possible to get very impressive results about microstructure evolution near the fatigue crack and below the wire surface after superelastic/ thermal cycles. High quality experimental results enabled a qualified analysis of the relationships between microstructure and properties of NiTi wires. The thesis consists of 118 pages, 35 figures in the theoretical part and 44 figures in the experimental part. The references used (140) reliably reflect the state of the art of the issue. The author participated in 12 published papers related to the dissertation. Most papers have been published in high quality professional journals (International Journal of Fatigue, Shape Memory and Superelasticity, International Journal of Plasticity, Acta Materialia etc.). Ing. O. Tyc is the first author in 5 papers focused on fatigue of superelastic NiTi wires.

The theoretical part of the dissertation clearly and concisely describes the state of the art. To this part of the thesis I have got only one minor comment. On page 18: "orientation relationship: $(001)_{Ni4Ti3}//(111)_{B2}$, $[010]_{Ni4Ti3}//[213]_{B2}$". Directions have to lie in the corresponding planes. It should be $[21\bar{3}]_{B2}$ or $[\bar{2}\bar{1}3]_{B2}$.

The experimental approach is based on thermomechanical tensile tests and fatigue tests in a wide temperature interval made on polycrystalline NiTi wires with the controlled grain size. Defined microstructure was obtained by pulse heat treatment of the cold worked wire resulting in the grain size in the interval from 20 to 250 nm. Furthermore, nickel rich NiTi wires with the grain size of 75 nm were subjected to annealing at 400 °C for 1 hour in order to promote precipitation of Ni_4Ti_3 particles. The NiTi wires with the finest B2 grains (20 nm) displayed a stable stress-strain response but the lowest fatigue life, while the wires with the coarsest B2 grains (250 nm) showed up an unstable response and the highest fatigue life, though the number of cycles to rupture decreased rapidly with increasing test temperature.

Based on the experimental results a suitable strategy how to improve the fatigue behaviour of NiTi wires in the superelastic regime in tension has been proposed: to create microstructure that allows for some plastic deformation while supressing microstructure refinement. A scheme allowing for estimating the instability of functional behaviour of various NiTi wires in a wide range of thermomechanical loading tests was introduced.

The formal level of the thesis meets high standards. I have a little comment on readability of details in some figures (for example Figs. 3, 19 and 33 in the theoretical part). Some more comments:
Page 50: Captions to Fig. 3 e,f: "the stress-strain curves were recorded in uniaxial tensile tests at 20 °C". However, corresponding graphs are missing.
Page 69: Comment to Fig. 19: "austenite twins extending across whole grains, sometimes forming wedges and/or crossing over grain boundaries". Could you explain what do you mean with crossing of austenite twins over grain boundaries?
In the discussion on the results achieved I would like to ask a few questions:

1. I understand that experimental material was supplied by Fort Wayne Metals. What was the content of interstitial elements in NiTi wires investigated?

2. It is no doubt that the quality of surface and subsurface layers plays a critical role in the nucleation stage of fatigue damage. Did you check NiTi wires in the as-received state in terms of occurrence of short defects across/around TiC particles and what was the size distribution of these particles? It is very likely that these particles also play a very negative role during cold forming processes. Are there any approaches how to reduce the roughness of thin NiTi wires after cold forming?

3. What is the repeatability of the pulse heat treatment results (in terms of grain size)? What parameters must be taken into account at process optimization?

Ing. Ondřej Tyc submitted the Dissertation Thesis at a high professional level which meets all the requirements for this type of qualification works. He proved the ability to perform sophisticated experiments and correctly evaluate the results obtained. I do recommend the Dissertation Thesis for the defense and in case of the successful defense to award the title Doctor of Philosophy (Ph.D.).

prof. Ing. Vlastimil Vodárek, CSc.

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