SUPERVISOR´S JUDGMENT OF THE PH.D. DISSERTATION

Author: **Ondřej TYC**

Title: **Effect of microstructure on fatigue of superelastic NiTi wires**

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Aim, methodology, and results of the thesis

The functional and structural fatigue performance of superelastic NiTi shape memory alloy (SMA) is one of the key issues to be solved in the SMA research. The main objective of the thesis was to reveal the links among functional fatigue, structural fatigue of NiTi wires, virgin austenitic microstructure, and test temperature. In addition, microstructure evolution during thermomechanical (especially superelastic) loading at various conditions (temperature and stress) was taken into consideration, which is a novel approach in the SMA field.

The tested NiTi wires were heat-treated by pulsed electric current and also conventionally annealed in an environmental furnace to adjust the microstructure as desired. Low cycle fatigue tests in the superelastic regime were performed to characterize functional properties and structural fatigue of the prepared NiTi wires with specific microstructures. NiTi wires having different nanocrystalline microstructures were subjected to thermomechanical tensile tests and fatigue tests in a wide temperature range. Accumulated unrecovered strains and microstructures evolving upon cycling were analyzed. The tensile cycling tests were complemented by extensive electron microscopy (TEM and SEM) analysis of the virgin and thermo-mechanically loaded samples to evaluate changes in microstructure brought about by tensile cycling.

If the stress-induced martensitic transformation in the cycled wire proceeded at low stress (< 100 MPa), the cyclic responses were stable (only marginal accumulated unrecovered strain and a few isolated dislocation loops and segments). If the forward and/or reverse martensitic transformation proceeded under large external stress (> 200 MPa), the responses of samples with 250 nm grain became unstable (large accumulated unrecovered strains and high density of dislocations and deformation bands). A scheme allowing for estimating the cyclic instability of functional behaviors of various NiTi wires in a wide range of thermomechanical loading tests was proposed and published. NiTi wires with the smallest grains (~ 20 nm) displayed a stable cyclic stress-strain response but the lowest fatigue life ($N_f < 2500$ cycles), while the wires with the largest grains (~ 250 nm) displayed an unstable cyclic stress-strain response and highest fatigue life ($N_f \sim 12000$ cycles), though the N_f decreased rapidly with increasing test temperature. A suitable strategy to improve the fatigue performance of superelastic NiTi is proposed in the thesis. It consists of the idea to create a special microstructure that allows for some plastic deformation while suppressing the formation of deformation bands/twins leading to microstructure refinement. Among the tested microstructures, these requirements are best matched by the aged wire containing 75 nm grains and Ni_4Ti_3 nano-precipitates.

Assessment of the dissertation and the student

The scope of the dissertation required a complex approach. The candidate, well educated in stress-strain analysis through his master studies, had to deepen his knowledge in scanning and transmission electron microscopy imaging methods. He benefited from the unique instrumentation available in the Institute of Physics but he also learned how to develop further the use of these instruments requiring skills in mechanics, electronics, and LabVIEW software. He thoroughly and independently carried out a large number of fatigue experiments and electron microscopy observations. He has proven to be able to design experiments based on the latest information from the literature.

The dissertation has in total 120 pages, it is written in a classical scheme: State of the art, Objectives of the dissertation, Experimental details, Results, Discussion, and Conclusions. It should be pointed out that the list of author's publications related to the dissertation has in total 12 items, mostly papers in respected international journals, proving the ability of the author to collaborate with colleagues. Ondřej Tyc is the first or corresponding author of 5 papers, which is showing his competence to write and publish results of high-quality research.

In our opinion, the thesis is written clearly and succinctly. The candidate is self-reliant, reliable, and competent. During the thesis, he made great progress in electron microscopy, fatigue testing, and writing papers.

The presented work satisfies, without any doubt, all the requirements laid on a Ph.D. thesis. It contains numerous original results, most of them already published in respected journals. The graphical layout, as well as the language level, are very good. All the aims were fulfilled. It is our pleasure to recommend this Ph.D. thesis to proceed for public defense.

Prague, August 30, 2021

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Petr ŠITTNER Miroslav KARLÍK