Supervisors´ judgment of the Ph.D. dissertation

Author: Ing. **Lukáš Kadeřávek**

Title: **MULTISCALE STRAIN LOCALIZATION IN THERMO-MECHANICAL FATIGUE OF NiTi SHAPE MEMORY ALLOYS**

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Aim, methodology and results of the thesis

The aim of this dissertation was to propose ways how fatigue performance of commercial NiTi can be possibly improved based on the results of dedicated experiments focussing on the role of macroscopic strain localization phenomena in cyclic tensile deformation of thin NiTi wires and sheets. Given the complexity of the topic of fatigue of phase transforming shape memory alloys, this is a very ambitious goal. Intuitively, one would expect that strain localization phenomena worsen the fatigue performance of SMAs, particularly in the case of partial superelastic tensile cycling. It was not known whether this is the case also in complete superelastic cycling at constant temperature or in thermomechanical actuator cycling. It was known that NiTi tends to deform in a localized manner in the tensile test when it undergoes stress induced cubic to monoclinic transformation. However, it was unclear whether it is also the case for martensite reorientation or plastic deformation or in thermally driven tensile deformation in actuator tests. The role of strain localization phenomena in NiTi fatigue was completely unknown when the work on this dissertation started in 2016. Moreover, strain localization can be observed in NiTi on nanoscale, mesoscale and macroscale and it was not known how if these localization phenomena are cross linked over the scales (and how).

Ing. L. Kadeřávek addressed this ambitious aim mainly from the experimental point of view and obtained new results going beyond the state of the art in the SMA field. The dissertation is organized in a traditional way with an Introduction and State-of-the-art chapters introducing the problem to be solved, results presented and discussed in four chapters (3-6) and a Conclusion chapter summarizing the obtained results. Results reported in chapters 3 and 4 inform mainly on the used experimental methods and NiTi samples, respectively. Chapter 5 deals with the strain localization phenomena in monotonic tensile testing of NiTi wires. It is very interesting to learn about the effects of the test temperature, strain rate and virgin austenitic microstructure on strain localization phenomena in NiTi. Very interesting and completely new are the results reported in chapter 5.3, particularly to the deformation mechanism of the ultrafast necking or propagation of Luders band front with 40% strain localized in the front. Chapter 5.4 would deserve more theoretical analysis to be performed, particularly in the evaluation of the EBSD studies. Chapter 6 contains key results of the Thesis dealing with functional and structural fatigue of NiTi SMAs phase transforming during cyclic thermomechanical loads. Again, it is very interesting to realize the consequences the strain localization phenomena have for NiTi fatigue. It is exciting to read about 200000 superelastic cycles till failure achieved using commercial NiTi #1 medical grade wire just optimizing the testing conditions and materials state of the wire. Chapters 6.6 and 6.7 are rather brief, the topics would deserve more systematic experimentation and analysis of the obtained results. Conclusions are brief and clearly formulated.

Assessment of the dissertation and the student

As introduced above, the aim of the dissertation required the application of a method which would enable in-situ tracking of local strains in NiTi thin wires and ribbons (20-500 μm), subjected to thermomechanical loading tests. This can be regularly achieved on flat sample deformed at room temperature by the digital image correlation (DIC) method. However, for application of the DIC method to 20 μm thin NiTi wires deformed in the tensile test from -100 to 500 °C, upon thermal cycling under applied stress or during necking when the localized deformation occurs within a fraction of a second, adaptation of the methods had to be developed including hardware, LabView software, pattern evaluation software, application high-speed optical cameras etc.

Besides this, Ing. L Kadeřávek designed and built a unique thermomechanical loading tester including special environmental chambers for in-situ optical and x-ray diffraction experiments, LabView control and data acquisition software. He benefited from the unique instrumentation available at the Institute of Physics but he also learned how to develop further and maintain these instruments. He designed the hardware using CAD software, made personally some components, acquired basic skills in electronics, programming and control necessary for developing and using sophisticated thermomechanical loading testers.

Ing. L Kadeřávek co-authored 21 highly cited publications in impacted journals as for example Progress in Materials Science, Acta Materialia or Applied Materials Today, where his role typically consisted in performing thermomechanical loading experiments on SMAs. Although the share of his work on those articles is essential, the research reported in these articles, except for the article [1] (Kadeřávek et al *Shape Memory and Superelasticity*, (2023), 1-19), were not included into the Dissertation. This shall be appreciated since it is not common in the history of the team he has joined (most of Ph.D. students tend to include the published work of the team in the thesis). Because of that, most of the results in the Dissertation shall yet to be published.

Ph.D. study of L Kadeřávek lasted relatively long (8 years) due to two reasons. He graduated from the Faculty of Engineering at the Czech University of Life Sciences Prague where he studied topics such as road transportation and city traffic. Hence he had to spend a lot of his time on theoretical study of fields such as solid state physics, deformation physics, crystallography. The second reason is that during his Ph.D. study L Kadeřávek always actively collaborated with his colleagues on the research, which has not been included in the thesis. For example, he did most of the background experimental work for the preparation of x-ray diffraction experiments in ESRF as well as the work on performing the experiment on the beamline in Grenoble. He is a co-author of highly cited articles written from that research [3,4]. But these results do not appear in the Dissertation. L Kadeřávek also performed thermomechanical loading experiments reported in modelling works [5,7,15,18] but did not use these results in his thesis.

During the course of his Ph.D. study, Ing. L Kadeřávek has become an expert in thermomechanical testing of SMAs. Moreover, he has learned several methods as for example in-situ testing in SEM, EBSD, electrochemical testing, etc. He thoroughly and independently carried out very large number of fatigue experiments. He has proven to be able to design experiments based on the latest information from the literature.

The dissertation has in total 124 pages, the list of author's publications related to the dissertation has in total 21 items, mostly papers in respected international journals which proves the ability of the author to carry out research in collaboration with colleagues. Ing. L Kadeřávek is the first author of 1 article, which proves his ability to write and publish high quality research results in scientific journals.

In our opinion, the thesis is clearly organized and written. The candidate is self-reliant, reliable and competent. The work presented in the Thesis satisfies all the requirements laid on a Ph.D. thesis. It contains a large number of original results that yet need to be published. The graphical layout as well as the language level are acceptable. All the aims were fulfilled. It is our pleasure to recommend this Ph.D. thesis to proceed for public defence.

Prague, March 28, 2023

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