Opponent´s opinion

of the Dissertation Thesis

Multiscale Strain Localization in Thermomechanical Fatigue of NiTi Shape Memory Alloys

Author: Ing. Lukáš Kadeřávek, Czech Technical University in Prague, Faculty of Nuclear Sciences and Physical Engineering, Department of Materials

Degree Programme: Application of Natural Sciences

Field of Study: Physical Engineering

Supervisor: prof. Dr. Ing. Petr Haušild, Department of Materials, Faculty of Nuclear Sciences and Physical Engineering – CTU, Prague

Supervisor Specialist: RNDr. Petr Šittner, CSc., FZU Institute of Physics of the Czech Academy of Sciences, Prague

Unique shape memory properties (both mechanical and thermal memory) of NiTi alloys have been utilized in many engineering and medical applications. However, some applications are severally limited by the low fatigue life of components made of these alloys. There is no doubt that the main topic of the dissertation, which can be formulated as "how the fatigue properties of commercial NiTi alloys can be improved", is very useful and current.

The theoretical part of the dissertation thesis clearly and concisely describes the state of the art of the issue. To this part of the thesis I have got only one minor comment. Page 9: " ..the quenching process of carbon steels which induced large volume variation inside the microstructure, thus creating many oriented defects and significantly hardening the microstructure." The main contribution to hardening of martensite in carbon steels is related to carbon atoms in octahedral positions of martensite.

The thesis consists of 124 pages, 9 figures in the theoretical part and 78 figures in the experimental part. The references used (122 in total) reliably reflect the state of the art of the issue. In the period of 2015 – 2023, the doctoral student participated in 21 published papers related to the topic of the dissertation. Most papers were published in high quality professional journals (International Journal of Fatigue, Shape Memory and Superelasticity, International Journal of Plasticity, Acta Materialia, Materials and Design, Materials Science and Engineering A, etc.). Ing. Lukáš Kadeřávek is the first author in one paper focused on localized plastic deformation of superelastic NiTi wires in tension.

The results of a very extensive set of experimental activities are presented in the thesis. The effect of test temperature and strain rate on strain localization during monotonic tensile testing of NiTi wires was studied. Subsequently extensive studies of the effect of superelastic tensile cycling were carried out. The effect of conventional heat treatment and fast pulse treatment of commercial NiTi alloys on fatigue properties was investigated. An unidimensional digital image correlation (1D-DIC) method has been developed since the strain localization is very important for the fatigue performance of NiTi alloys. Systematic application of the 1D-DIC method made it possible to get a better understanding of the phenomena controlling strain localization, accumulation of damage and fatigue performance of NiTi shape memory alloys. Application of this method made it possible to get results which are beyond the state of the art in the given field. It was demonstrated that in addition to the localized deformation of NiTi via stress induced martensitic transformation, localized plastic deformation can occur also during the plastic deformation of the wire in the martensite phase. It was observed that plastic deformation associated with propagation of macroscopic Lüders bands enabled unusually large plastic strain (up to ~ 40 %). Mechanical tests were accompanied by a sophisticated post-mortem TEM analysis. Plastic deformation of monoclinic martensite in NiTi was explained by a combination of deformation twinning and dislocation slip kinking; this mechanism was termed as “kwinking”.

A very long fatigue lifetime of commercial NiTi wire was achieved in superelastic tensile cycling with adaptive limits at temperature of 10 °C (Nc > 200 000 cycles). Let´s hope this significant improvement will be confirmed statistically.

The 1D-DIC method is appropriate for investigations in NiTi wires since cone shaped Lüders bands propagate along the length of samples during the tensile tests. The thesis also includes the results of the application of the 2D-DIC method to analyse the effects of stress concentrators on the superelastic cyclic tensile deformation of flat samples in the form of a dog bone made from thin sheets. It was demonstrated that the character of stress concentrators (notch, circular hole, elliptical hole) significantly affects strain localization phenomena and the number of cycles until failure in superelastic fatigue tests of NiTi alloys. Larger stress concentrations around these stress concentrators lead to a faster accumulation of local unrecoverable strain. An attempt was made to study stress induced martensitic transformation in NiTi tensile samples using in situ EBSD investigations. The spatial resolution of this technique limits characterization of fine microstructure in this material.

In the final chapter some experiments focused on the fatigue of NiTi alloys in thermomechanical actuators are presented. This research is still in progress.

The formal level of the thesis meets high standards. I only found a few grammatical imperfections, e. g. Kroll´s reagent (page 30), via newly discovered deformation mechanism (Conclusions, point 5).

I have got some comments and questions which could be discussed during the defense of the thesis:

- Page 44: Fig. 23d is not discussed in the text and from the caption it is not clear what part of the fatigue sample these micrographs represent. Could you comment on it briefly?
- Page 52: Ring diffraction patterns in Fig. 29 demonstrate that interplanar distances of planes $\{011\}_A$ and $(-111)_M$, $\{002\}_A$ and $(022)_M$, $\{112\}_A$ and $(113)_M$ are very close each to other. How did you find out that discontinuous rings in Figs. 29c and 29d contain spots from both phases?
- Page 55: " …crystalline and nearly amorphous lattices.." What do you mean with "amorphous lattice"?
- Page 97: The caption is missing for Fig. 73d.
- Page 108: " In the case of the wire deformed at 10 °C, one lamellae was cut from the middle part of the fractured wire." However, Fig. 58b shows that the cyclic deformation at 10 °C was stopped by the operator after 200 000 cycles.
- Page 108: Experimental material in Fig. 87 is reported as NiTi#5. This name of experimental material is not specified in Table 2.
- In some cases R phase was observed in NiTi#1 fatigue samples. Did you get an experimental evidence that in samples containing R phase Ni_4Ti_3 precipitates were present?
- TEM SAED analysis of thin foils cut in areas of high local strain is a hard task: the minimum size of the selected aperture is limited, diffraction spots corresponding to individual phases can be very close each to other, microstructure is very fine and contains a lot of defects. Dark field images should be interpreted only in the area selected by the selective aperture. For example, in Figs. 32h and 32i there are DF images taken using martensite spots belonging to one zone axis (Fig. 32d).

Interpretation of fine bright areas in Fig. 32h out of the selected area (Fig. 32a) may not be unambiguous. It is difficult to follow the indexing of diffraction patterns in Figs. 32c and 32d (faint spots). It might be better to use inversed contrast of diffraction patterns.

The results presented in the dissertation thesis prove that the planned goals of the work were fulfilled. Ing. Lukáš Kadeřávek 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.
Department of Materials Engineering and Recycling
Faculty of Materials Science and Technology
VŠB-TU Ostrava

Ostrava, 20. 5. 2023