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Reviewer's report on a Ph.D. thesis

INFRARED CALORIMETRY APPLIED TO THERMOMECHANICAL CHARACTERIZATION OF NiTi ALLOYS

by Antoine JURY, Czech Technical University in Prague

supervisors: prof. Dr. RNDr. Miroslav Karlík (Czech Technical University), prof. Dr. Xavier Balandraud (Université Clermont Auvergne).

reviewer: doc. Ing. Hanuš Seiner, Ph.D., DSc.

The thesis summarizes the candidate's work in the field of heat source reconstruction and thermomechanical analysis of superelastic polycrystalline NiTi wires. The topic of the thesis is interesting from the fundamental research point of view (merging basic thermodynamics with numerical modeling and advanced experimental characterization), as well as regarding the application potential of NiTi wires.

Main results of the thesis were already accepted for publication in quite highly-ranked journals in the field of mechanics of materials (*Experimental Mechanics* and *Materials & Design*), which confirms the quality and international-level impact of the work. The candidate also presented his work at several conferences, proving his ability to communicate the research to the SMA-oriented community.

The thesis consists of three main parts, the first being the review of literature knowledge, the second comprising results on heat localization in NiTi wires in a single superelastic cycle, and the third dedicated to fast characterization in cycled samples. I highly appreciate the first part, spanning over more 40 pages and giving a very good review. The material properties, the experimental techniques and the theoretical models are all described in sufficient detail and the text is logically structured.

When the thesis enters the parts of the candidate's own research, the text goes more technical, and gets slightly harder to read. The formal and language quality remains quite high, but I feel that sound and summarizing conclusions (not just listing the main features of the observed behavior) are sometimes lacking. This is the only reservation I have regarding Chapters 2 and 3; otherwise, these parts are carefully prepared and report clearly on the obtained results. From them, I find those concerned with the partial localized → delocalized behavior upon cycling (Figure 3-10 and below) as the most interesting. The relation of this behavior and the shape of the stress-strain curves in Figure 3-8 (i.e., its evolution with the number of cycles) remains to be resolved to some extent; however, it turns out that involving the 0D (delocalized) approach here was a fortunate choice and brought very valuable findings.

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Regarding the research reported in Chapters 2 and 3, I have the following questions I would like the candidate to answer within his defense:

1. In Table 2, page 58, the lowest thermal diffusivity as obtained for oriented martensite. All literature data for self-accommodated martensite give higher numbers. This is a surprising finding for me, as I would expect the high density of defects (twin walls) in self-accommodated martensite to hinder the heat transfer. Do you have any explanation for that? May anisotropic heat conductivity in monoclinic B19' play some role here?

2. Page 68 and around, is it really correct to assume the merging points as localized sources/sinks of heat? In my understanding, they are just points were two interfaces meet, both carrying some moving heat sources, so their contribution adds. Or is there any non-linear phenomenon going beyond this simple superposition?

3. What are the physical/microstructural processes behind the WHSs? There might be some R-phase transformation for the forward transition, but what happens in martensite? Or are the WHS for the cycled sample just localized events that fall below the resolution limit of the temperature mapping? I know that the thesis touches the problem here and there, but I was not able to find any sound opinion on this.

4. On page 89, NiTi is claimed to be an excellent candidate for elastocaloric systems, which is undoubtedly true. However, the latest trends in the solid-refrigeration in SMAs go rather in the direction of Heusler NiMn-based alloys (NiMnIn, NiMnSnCu, NiMnGaIn) or similar NiFe(Ga)-based. Can you compare the solid-refrigeration performance performance of NiTi and these materials?

The thesis concludes with an (unnumbered) chapter entitled *General conclusions and perspectives*. It brings a clear and well-structured summary of the results and some perspectives for further work. Regarding the perspectives, I would appreciate a more physics-based (or technology-driven) future outlook, while the candidate here lists mostly technical details that remain to be explored in more detail. Nevertheless, the section as a whole proves the candidate's deep insight into the topic and serves as an appropriate ending to the thesis.

Regarding the formal issues, I found only a very small number of misprints or typos. Also, the graphical level of the thesis is quite high. In some places, the thesis might deserve some additional polishing for example:

- subplots c) to f) in Figure 2-1 are quite hard to understand before the reader reads the whole subsection
- several figures in the first chapter are reprinted with questionable quality (pages 18, 24, etc.)
- some characters in the equations should not be italicized (for example the "d" in total derivatives)
- some wording is chosen unfortunately, for example "analyze" is a verb but is used as a noun; the word calorific should be used rather for a fuel, food or chemical reaction, etc

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These are only very minor issues that do not lower the level of the thesis.

To conclude, I can say that the research reported in the thesis is **highly topical** and **contributes significantly** to the field of NiTi fatigue and fracture. The personal contribution of the candidate is **obvious and essential**. The used **methods and approaches were chosen properly**, and enabled the **aims of the thesis to be completely fulfilled**. I find that the **thesis fulfills all legal requirements (§ 47 sect. 4) for Ph.D. dissertations, and I recommend the thesis to be advanced for defense and the candidate to be awarded the degree.**

In Prague, May 25, 2021

Hanuš Seiner