Review report on the PhD thesis

Thesis title: Infrared calorimetry applied to thermomechanical characterization of NiTi alloys

Candidate: Antoine JURY

For further developing important engineering applications and better understanding the special functions such as super-elasticity, shape memory effect and elastocaloric effect, the thermo-mechanical coupling in NiTi Shape Memory Alloy (SMA) are intensively studied in literature, from microscopic martensitic phase transformation mechanism to macroscopic recoverable deformation. Based on the previous research works about "deformation calorimetry", this thesis further develops the method of "infrared calorimetry" with the technique "Heat Source Reconstruction" (HSR) combining the infrared temperature measurement and the energy-balance equations to identify (trace) the heat sources in the meso-scale complex thermo-mechanical coupling that includes numerous nucleation/propagations/annihilations of Luders-band fronts—localized phase transformation releasing/absorbing latent heat. Moreover, Mr. Antoine JURY performed the experiments on trained and virgin NiTi specimens and applied the HSR method to analyse the heat sources in various situations in a systematic comparative study: the forward/reverse martensitic phase transformation, the homogeneous heat sources vs. localized heat sources, band-front nucleation vs. annihilation (merging), etc., which are very helpful in understanding and theoretical modelling on the macroscopic global stress-strain response. Particularly, this method has the potential to evaluate accurately the elastocaloric effect (the cooling capability) for solid-state refrigerator made from SMAs or other materials. Although the main body of the thesis including three chapters is quite short (less than 90 pages), the writing and organization are very good. Obviously, the thesis demonstrates a better thermo-mechanical characterization method for both academic research and engineering applications of the materials; it is a nice piece of work.

This thesis includes three chapters, one general introduction, one general conclusion, three appendixes and extensive references. The general introduction at the beginning of the thesis (pages 1-3) provides the basic background of the material's thermo-mechanical coupling and the associated characterization methods. Particularly, the motivation, the objective and the general methodology of the present study are clearly given.

Chapter 1 (pages 4-46) is a detailed literature review on different aspects related to the present study. Section 1.1 describes the fundamental physical origin of the special properties of NiTi SMAs and the associated stress-temperature phase diagram summarizing the various loading paths for the martensitic phase transformation to demonstrate the different phenomena such as Super-elasticity, Shape Memory Effect, Damping and elastocaloric effect. Section 1.2 reviews different deformation calorimetry methods and points out their advantages/disadvantages and difficulties/problems. Section 1.3 summarizes the key mathematical expressions of the thermodynamic relations of the thermo-mechanical fields/parameters, which help develop the technique to characterize the heat sources, so-called "Heat Source Reconstruction" (HSR) whose main assumptions and operation principles are introduced in Section 1.4. This well organized literature review clearly indicates the feasibility and uniqueness of the objective of the present study.

Chapter 2 (Pages 47-70) reports the experimental setup/procedures and the experimental results measuring the spatio-temporal temperature distribution and characterizing the heat sources caused by the deformation of a trained NiTi SMA wire under a typical force-controlled tensile loading-unloading superelastic cycle with the help of the infrared temperature measurement and the HSR technique (heat source reconstruction method). As the input parameters of the HSR method, several thermal material properties and heat-transfer conditions were measured with extra independent experiments (Fig. 2-1), such as thermal diffusivity (thermal conductivity) of the austenite and the detwinned martensite, the heat exchange rate between the specimen and the environment. The HSR method helped track the evolution of the localized stress-induced martensitic transformation which releases/absorbs latent heat. Particularly, the nucleation and the merging of the Luders band fronts during forward and reverse martensitic phase transformation were identified and the associated heat sources were quantified and compared with the "weak heat sources" of the homogeneous deformations before and after the localized transformation. Although there is no local deformation strain measurement in current experiments, this chapter demonstrates clearly that the method is able to identify accurately the evolving localized heat sources (the band fronts) to study the kinetics of the martensitic phase transformation under the complicated thermo-mechanical coupling situations.

Chapter 3 (Pages 71-89) reports the heat-source quantification of the 20 cycles of the stress-induced martensitic phase transformation in a virgin NiTi wire. During the 20 cycles, the thermo-mechanical states (strain and temperature) of the specimen evolved with the thermal and mechanical energy balances, leading to different temperature variation, different strain amplitude, different stress hysteresis and different associated heat sources in the cycles. From the characterization of the heat sources and their comparisons with the mechanical work/dissipation, some insights into the material's functional fatigue were obtained: a portion of mechanical energy was stored in the material in the forms of microstructure defects or/and residual martensite during the fatigue testing cycles. Moreover, it was also revealed that the functional fatigue influenced the kinetics of the phase transformation: the number of the events of the band front nucleation and merging increased with the loading cycles, i.e., when the specimen is quite fresh, only one or two band nucleation/merging; by contrast, when the specimen is well trained, numerous bands were simultaneously nucleated/annihilated, globally like homogeneous deformation or called "delocalization of martensite phase transformation" in the thesis. Taking functional fatigue as an example where both mechanical work/dissipation and heat source are coupling and evolving, this chapter shows the strength of this method in decoupling and quantifying the thermo-mechanical quantities.

Finally, in the general conclusion (Pages 90-92), the novelties, key conclusions and possible future developments of the present study are summarized. The three appendixes containing technical details and the list of 172 references are at the end of the thesis.

To conclude this review, I think that Mr. Jury has submitted a well-organized thesis, which reflects his good understanding on the topic and his outstanding contributions to developing the powerful method for the material research. Therefore, I propose the acceptance of this thesis.

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Yongjun HE
Maître de conferences (HDR)
Institute of Mechanical Sciences and Industrial Applications (IMSIA)
ENSTA-Paris, Institut Polytechnique de Paris (IP Paris)
828 Blvd des Marechaux, 91120, Palaiseau, France

Email: yhe@ensta.fr