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Thermomechanical aspects of the nucleation and development of stress-induced martensitic transformation in shape memory alloys

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Due to the dynamic development of technology and the increasing demand for materials, as well as growing environmental awareness, particular interest has recently been focused on multifunctional smart materials, able serve various purposes, such as working, sensing, and crack-healing. This group includes shape memory materials: alloys, polymers and composites. Among these, shape memory alloys (SMAs) have attracted an especially high level of attention because of their unique properties, such as the shape memory effect and superelasticity, related to large recovery strain, stress, and energy storage and dissipation, which are important for their application as actuators, elements of dampers, or earthquake protection systems. The SMA properties are caused by a stress- or temperature-induced crystallographically reversible martensitic transformation between a high-symmetry austenite and a low-symmetry martensite structure, related to changes in shape and size. These properties, together with high sensitivity to temperature, allow the SMA to combine sensor and actuator functions, thus enabling miniaturization and various applications in biomedicine (cardiosurgical stents, guidewires, orthodontic braces), aircraft (joining elements, morphing), spacecraft (automatic doors), and the car and housing industries (sensors and actuators, conditioners, overheating protectors). In order to design shape memory elements for an application, thermomechanical properties are very important (Tobushi *et al.*, 2013). Such research has been carried out for years at the Institute of Fundamental Technological Research (IFTR): thermodynamic description by B. Raniecki, and A. Ziółkowski, micromechanics by H. Petryk and S. Stupkiewicz, as well as the experimental approach coordinated by W. K. Nowacki and L. Dietrich in collaboration with their Japanese partners S. Miyazaki and H. Tobushi. The results obtained using an infrared camera have turned out to be especially interesting, including for researchers from other centers. The high sensitivity (0.025K) and frequency (538Hz) of the camera, and most importantly the long tradition and extensive experience of the IFTR in experimental research on thermomechanical couplings in metals, all contributed to unique results on SMA properties being obtained.

The goal of the research presented here was to investigate the nucleation, development, and saturation of stress-induced martensitic transformation based on the effects of thermomechanical couplings. To this end, TiNi shape memory alloy was subjected to tension at various strain rates on the testing machine while a fast and sensitive infrared camera recorded the infrared radiation emitted by the SMA specimen, enabling its temperature to be calculated and the temperature distribution on it to be obtained.

Figure 1 shows an example of the stress and average temperature change vs. strain curves obtained during the TiNi SMA complete loading-unloading tension cycle, accompanied by infrared imaging. It was found that the initial, macroscopically

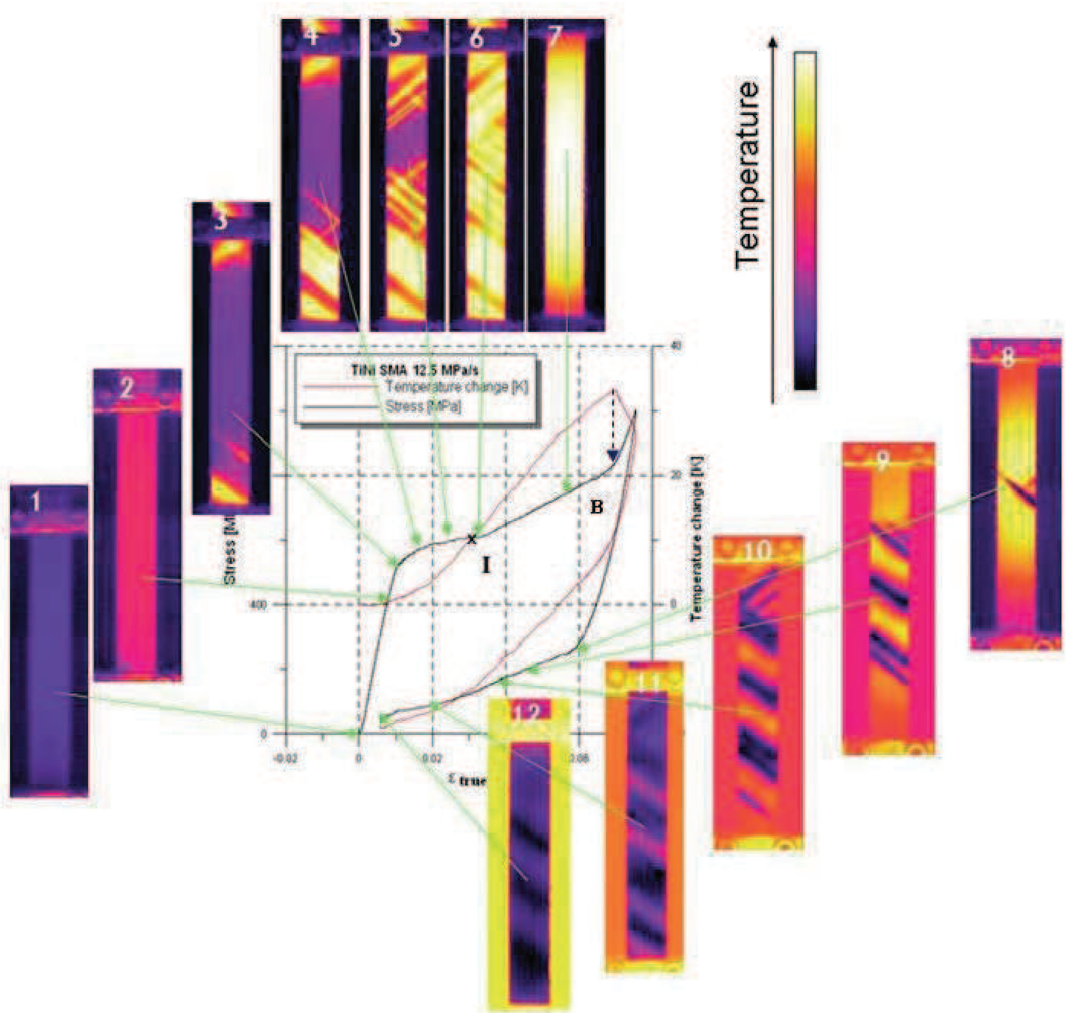

Fig. 1. Stress (black line) and temperature change (red line) vs. strain curves obtained during TiNi SMA tension at stress rate 12.5 MPa/s. The cross mark x indicates inflection point *I*, point *B* the transformation saturation stage. Thermograms show respectively: (2) initial uniform transformation, (3-7) macroscopic bands, related to exothermic forward, and (8-12) endothermic reverse transformation (Pieczyska *et al.,* 2013)

homogeneous transformation initiates even before the knee of the stress-strain curve and occurs at the same stress level for all strain rates applied. However, the stress of the localized martensitic transformations depends on the strain rate. The higher the strain rate, the higher the sample temperature due to the exothermic martensitic transformation and almost adiabatic test conditions. These follow a higher stress and more dynamic transformation process, revealed in the creation of numerous fine transformation bands, distinguished by different temperatures from the other part of the specimen (Figs. 1, 2). Moreover, an inflection point is noticed on the stress-strain curve, dividing the transformation range into two stages: the first macroscopically heterogeneous, when the bands nucleate and evolve throughout the specimen; the second, when the

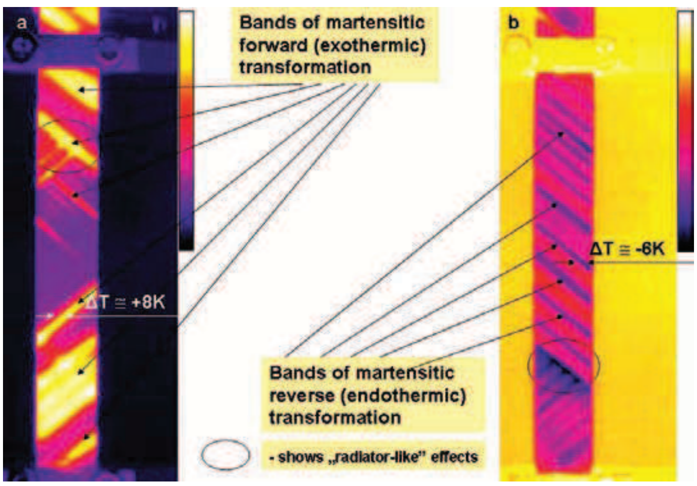

Fig. 2. Infrared imaging of two generations of macroscopic transformation bands in TiNi SMA during loading (a) and unloading (b) at stress rate 25 MPa/s (Pieczyska *et al.,* 2013)

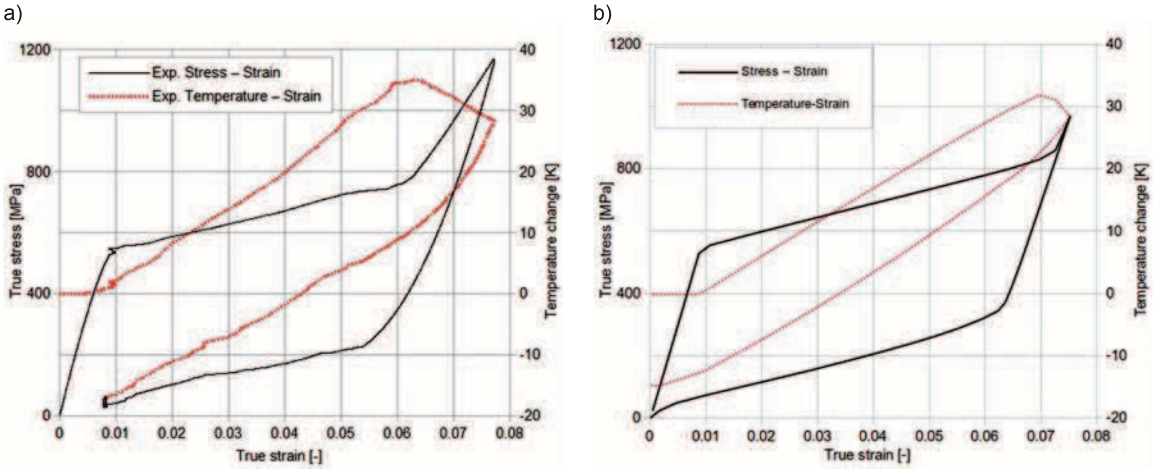

Fig. 3. Comparison of stress and temperature vs. strain curves for TiNi SMA during tension at stress rate 25 MPa/s: a) experimental, b) numerical results (Dunic *et al.*, 2014)

bands overlap, causing significant temperature and stress increase.

The macroscopic martensitic transformation bands obtained in infrared were confirmed using the digital image correlation (DIC) strain map technique by Daly *et al.* (*Acta Mater.* 55; 2008), and the authors refer to the results of Pieczyska *et al.* (*Bull. Pol. Ac., Tech.*; 52-3; 2004). However, the advanced infrared technique enables obtaining more details to be obtained. Namely, it has been shown in recent papers that at the advanced stage of the martensitic forward/reverse transformation, a new generation of much thinner bands can appear. They probably develop in those parts of the specimen where the need arises to compensate for the local stress-strain state instability, caused by the current loading stress-strain state and the transformation progress (Fig. 2).

Moreover, in the final part of the SMA loading, a decrease in the specimen temperature reveals the saturation stage of the exothermic transformation (Figs 1, 3).

It was also observed that nucleation of the localized martensitic forward transformation takes place in the weakest area of the SMA specimen, whereas the reverse transformation always initiates in the central part of the specimen (Fig. 1).

The effectively modified constitutive model proposed by Lagoudas has been implemented in structural finite element method (FEM) software and thermomechanically coupled with heat transfer FEM software in a partitioned approach. The research was performed by V. Dunic from Serbia, who found the SMA experimental data obtained at the IFTR on the Internet and successfully applied to EC project KMM-VIN for a vacation internship. The experimental results of the stress and temperature changes are quantitatively and qualitatively reproduced by the proposed numerical model, as shown in Fig. 3 (Dunic *et al.,* 2014).

It was also found that after the unloading, the SMA temperature decreases to below its initial temperature, quite significantly at lower strain rates (14 K; Fig. 3). This thermal effect, described by Pieczyska *et al.*, 2013, 2014, and important for another kind of SMA application (as cooling elements), is also confirmed by the proposed model (Fig. 3b).

The results obtained confirm the high accuracy of the equipment and the correctness of the applied experimental and modeling methodology, and aid understanding of the processes occurring in shape memory alloys in more detail, thus enabling their further application.

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Brain perfusion assessment using a time- and wavelength-resolved optical technique

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In recent years, a variety of optical techniques proposed for brain imaging have been extensively tested in clinical trials. One of the promising methods allowing for brain hemodynamics monitoring is near-infrared spectroscopy (NIRS). Photons of light from the near-infrared region can penetrate the studied tissue to a depth of up to 2 cm, so in measurements performed on the surface of the human head, the light can reach the gray matter of the brain. Changes in the concentrations of oxy- and deoxyhemoglobins (HbO_2 and Hb) can be estimated by considering the different spectral properties of these two forms of hemoglobin in the NIR region.

In NIRS measurements, light from a laser is delivered to a point of emission on the surface of the subject's head by an optical fiber. After penetrating the diffuse medium, a fraction of the photons emitted into the tissue return to the surface of the head, where they are collected at a distance of 2-4 cm from the point of emission and delivered to the photodetector. The changes in light attenuation measured at several wavelengths are used to calculate the changes in the concentrations of HbO_2 and Hb.

The NIRS technique has been successfully applied in the monitoring of brain oxygenation in neonates and infants. In such measurements the influence of the extracerebral tissue (skin, skull) on the signals measured is small. However, in measurements on adults this technique suffers because of the strong dependence of the optical signals on the changes in oxygenation of the extracerebral tissue. It has been reported that application of time-resolved NIRS is beneficial in these cases and allows for the separation of the signals originating from the brain by measuring the distribution of the times of flight of photons (DTOF) through the scattering object i.e. tissue.

The time-resolved method proposed in the Department for Biophysical Measurements and Imaging of the PAS Nalecz Institute of Biocybernetics and Biomedical Engineering (IBIB) is based on the use of an optical tissue monitoring methodology, combined with evaluation of the passage of a contrast agent, Indocyanine Green (ICG), injected into the forearm vein of the subject. Inflow of the ICG dye into the brain tissue leads to an increase in local absorption, and the fluorescence properties of the circulating dye can also be utilized (Gerega et al., 2012). The multichannel optical setup for time-resolved measurements developed in IBIB makes it possible to record the DTOFs at two wavelengths (Milej et al., 2013).

Analysis of the DTOFs is based on the derivation of changes in their statistical moments, which reveals different sensitivities to the absorption changes occurring at different depths in the tissue. It has been found that higher-order moments are selectively sensitive to changes in absorption of the cerebral cortex. This observation is the basis of the methodology allowing the determination of cerebral blood flow changes in the human brain by separating the components of the measured signals originating from the extra- and intracerebral layers of the tissue (Weigl et al., 2014).

The novel technical approach proposed recently by our group is based on time- and wavelength-resolved measurements of diffuse reflectance and fluorescence at multiple wavelengths from the near-