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# Thermomechanical investigations of martensitic and reverse transformations in TiNi shape memory alloy

E. A. PIECZYSKA<sup>1\*</sup>, S. P. GADAJ<sup>1</sup>, W. K. NOWACKI<sup>1</sup> and H. TOBUSHI<sup>2\*\*</sup>

Institute of Fundamental Technological Research, Polish Academy of Sciences, 21 Świętokrzyska St., 00–049 Warsaw, Poland
 Department of Mechanical Engineering, AICHI Inst. of Technology, 1247, Yachigusa, Yakusa-cho, Toyota-city, Aichi, 470-0392, Japan

Abstract. Shape memory alloys are characterised by interesting properties, i.e. shape memory effect and pseudoelasticity, which enable their increasing application. Thermomechanical aspects of martensitic and reverse transformations in TiNi shape memory alloy subjected to tension tests were investigated. The stress-strain characteristics obtained during the tests were completed by the temperature characteristics. The temperature changes were calculated on the basis of thermograms determined by an infrared camera. Taking advantages from the infrared technique, the temperature distributions on the specimen's surface were found. Heterogeneous temperature distributions, related to the nucleation and development of the new martensite phase, were registered and analysed. A significant temperature increase, up to 30 K, was registered during the martensitic transformation. The similar effects of the heterogeneous temperature distribution were observed during unloading, while the reverse transformation, martensite into austenite took place, accompanied by significant temperature decrease.

Keywords: shape memory alloy, pseudoelasticity, transformation front, thermomechanical, investigations.

#### 1. Introduction

When the shape memory alloy is seemingly permanently mechanically deformed at a temperature below a certain transition temperature and reverts to its original shape after heating at the temperature above the phase transition temperature, it is called a shape memory effect. Such phenomena can be applied to medicine, stomatology as well as during designing of robots, solid-state heat engines, etc. Pseudoelasticity is the material ability to be strained much above the elastic limit and return to its initial shape during unloading, showing a hysteresis loop. Such features, like nonlinear elastic behaviour, high yield stress or significant internal damping, lead to increasing application of these materials to actuators, damping elements or other "smart" structures. Since the elements are working in various conditions, the thermomechanical properties of the shape memory alloys are very important.

Experimental studies of the phenomena of initiation and kinetics of the phase transformation fronts were discussed in Shaw and Kyriakides [1], Shaw [2], Tobushi et al. [3] Lin et al [4], Gadaj et al [5, 6], Helm et al. [7], Pieczyska et al. [8, 9], and others. Significant progress was made in research concerning phase transformation behaviour in last decade. Nevertheless, the phenomena of the origin and further development of the new phase is still a subject of current studies. The attention of the present paper is focused on the aid provided by infrared thermography (IT) for investigation of the various thermomechanical aspects related to the phase transitions in TiNi shape memory alloy (SMA).

# 2. Theoretical background

According to the constitutive relationships proposed by Tanaka et al. [10] and Tanaka [11] and applied by Lin et al. [4] and Tobushi et al. [12], the deformation behaviour of SMA due to martensitic transformation can be described as follows:

$$\dot{\sigma} = D\dot{\varepsilon} + \theta T + \Omega\dot{\xi};\tag{1}$$

where  $\sigma$ ,  $\varepsilon$  and T represent the stress, strain and temperature, respectively. The coefficients D and  $\theta$  represent the modulus of elasticity and the thermoelastic constant, respectively. The quantity  $(\Omega/D)$  represents the strain range of the martensitic transformation. The internal state variable  $\xi$  represents the volume fraction of the martensite phase. In this way, the volume fraction of the parent phase is  $1-\xi$ . The dot over the symbols denotes the time derivative.

The transformation kinetics for the martensitic transformation can be described by the formula:

$$\frac{\dot{\xi}}{1-\xi} = b_M C_M \dot{T} - b_M \dot{\sigma} \geqslant 0 \tag{2}$$

and for the reverse transformation:

$$-\frac{\dot{\xi}}{\xi} = b_A C_A \dot{T} - b_A \dot{\sigma} \geqslant 0 \tag{3}$$

The material parameters:  $b_M$ ,  $C_M$ ,  $b_A$ ,  $\Omega$  and  $C_A$  can be found from the experiment.

The proposed equations enabled us to determine the fundamental conditions for progress of the martensitic and reverse transformations during the stress induced phase transformation, namely the direction of the stress and the temperature changes as well as to explain general principle of SMAs behaviour under various conditions.

<sup>\*</sup> e-mail: epiecz@ippt.gov.pl

<sup>\*\*</sup>e-mail: tobushi@aitech.ac.jp

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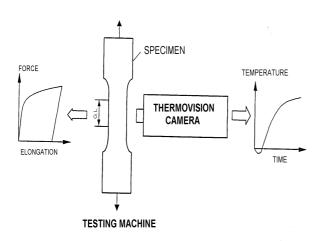




Fig. 1. Scheme and photograph of the experimental set-up designed for thermomechanical investigation of the stress induced phase transformations in shape memory alloys

# 3. Methodology

In order to study a thermomechanics of nucleation and further development of the stress induced martensitic transformation in TiNi SMA, providing accurate experimental data was necessary. To this end, strain controlled tension tests were performed with the quasistatic rate of deformation. A thermovision camera was used in order to register the distribution of infrared radiation and create thermograms that enabled us to calculate the temperature changes of the TiNi specimens subjected to loading. The infrared camera turned out to be very useful in the research concerning investigation of the stress induced phase transformations. It enabled us to identify the area of higher or lower temperature, related to occurrence of the new material phase, with high accuracy. The obtained temperature images were stored for further analysis; such approach allowed us to observe, register and analyze kinetics and thermodynamics of the phase transformations in shape memory alloys subjected to loading and unloading process.

## 4. Experimental procedure

The sheet specimens of  $160~\rm mm \times 10~\rm mm \times 0.4~\rm mm$  were cut from the band of SMA. Each specimen was covered before the testing by a thin layer of the candle carbon powder in order to obtain a higher and homogeneous emissivity, possibly stable during the deformation process, which in practice is not too easy to assure. The specimens were subjected to the strain controlled tension test with the strain rate equal to  $10^{-2}~\rm s^{-1}$ . The temperature changes were calculated as an average from the central specimen area  $\approx 400~\rm mm^2$  with accuracy up to 0.1 K. Scheme and photograph of the experimental set-up are shown in Fig. 1.

The applied research methodology is based on the experimental scheme like this. A specimen of the TiNi shape memory alloy (SMA), designed according to the program of investigation, was fixed in the hydraulic testing machine in order to obtain the stress-strain characteristics with a high accuracy. The infrared camera was used in order to find the temperature distributions of the material subjected to loading. It registers a distribution of infrared radiation from the specimen surface and creates thermograms with 50 Hz frequencies. A computer system of the data acquisition and conversion enables us to register, store and reconstruct the obtained mechanical and temperature data. In this way, the temperature changes can be presented as a function of various mechanical parameters in any time, as required.

## 5. Results

As results of the tensile experiments, the mechanical and temperature characteristics and the numerous thermograms showing temperature distributions on the surface of the specimens of the TiNi SMA were obtained (Figs. 3–8). The example of the worked out mechanical and the temperature curves during the complete loop of the phase transformation process was shown in Fig. 2:  $\Delta T$  denotes the average temperature calculated for the chosen specimen area while  $\Delta T_P$  denotes the temperature taken from the point where the first signal of significantly higher temperature related to the new phase was noticed.

Due to analysis of changes in the mean temperature of the sample surface one can draw suggestions concerning the courses of effects accompanying the strain induced phase transformation on a macroscopic scale, together with the influence of different factors upon those effects. One should remember, however, about an inhomogeneous nature of both the initiation and development, respectively, of phase transitions.

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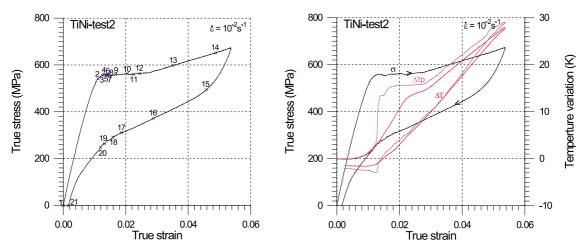


Fig. 2. Stress-strain curve and temperature changes of TiNi SMA:  $\Delta T$  denotes average temperature changes from the specimen area and  $\Delta T_P$  denotes temperature changes at the point where the phase transition start was noticed

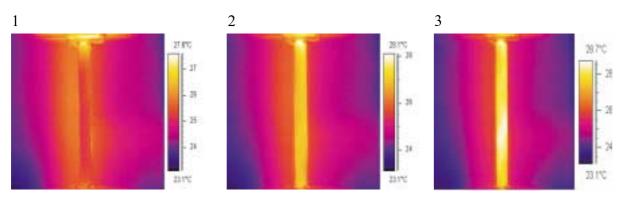


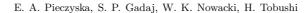
Fig. 3. Temperature distribution of TiNi SMA subjected to stress induced phase transformation during the initial part of loading. Numbers 1, 2, 3 correspond to the proper points of the stress-strain curve shown in Fig. 2, accordingly: 1 (before testing), 2 (initial elastic deformation),  $3 (1^{st} \text{ sign of in inhomogeneity})$ 

After the initial, elastic stage of deformation, a little overshoot followed by a stress plateau related to the initiation of the martensitic deformation was observed. During unloading, the reverse transformation (martensite into austenite) took place. It was observed that the both phase transformations were accompanied by significant temperature changes; the martensitic transformation was accompanied by the temperature increase up to 30 K, while the reverse transformation was accompanied by the decrease in temperature.

Taking advantages from the infrared camera, which monitored the specimen surface and registered the temperature distribution, it was observed that in initial range of the deformation, the temperature distribution on the surface of the examined specimen was uniform, which was pointed out on the homogeneity of the stress and the strain state along the specimen (Figs. 2, 3).

**5.1.** Martensitic transformation. Before the tension start the sample temperature was equal to the ambient temperature of 296 K. At the initial tension phase, i.e., as

the stress increases up to its local maximum (see Fig. 3) the specimen surface temperature increased, the thermal image, however, remained almost quite uniform indicating homogeneous nature of the deformation process. After the uniform temperature distribution registered in the elastic range of deformation, the temperature slightly increased in the central area of the sample (Fig. 3; the  $3^{rd}$  thermogram), which was followed by a sudden occurrence of the first inclined line of significantly higher temperature (Fig. 4; thermogram 4). This was the region of creation of the new, martensite phase. It occurred just at that moment when the overshoot registered on the stress-strain curve vanished, namely when the stress reached the local maximum (Fig. 2). A sudden increase in temperature, related to this phenomenon, was equal to about 6–10 K. After a while, a few such lines parallel to each other occurred towards the sample borders, as well as the next "family" of them, which was developed in the perpendicular direction (Fig. 4; thermograms 6–9). The lines, characterised by significantly higher temperature, immediately evolved into bands. The highest temperature, up to 30 K, was registered at the cross-section of the bands



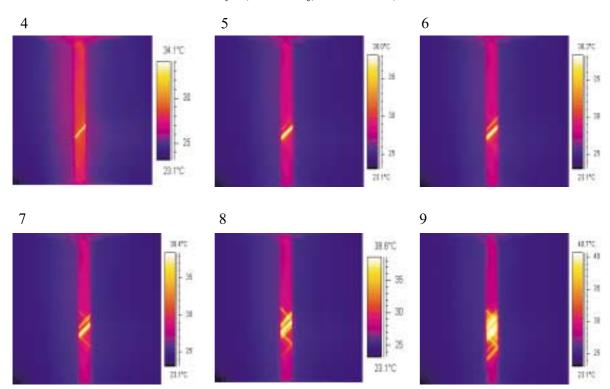


Fig. 4. Temperature distribution of TiNi SMA subjected to stress induced phase transformation during loading (martensitic transition). The numbers above the thermograms correspond to the proper points of the stress-strain curve shown in Fig. 2

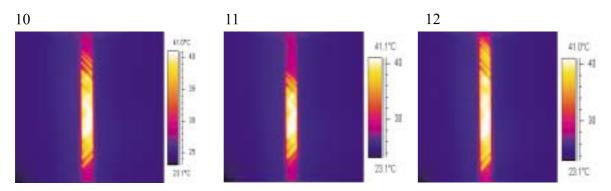


Fig. 5. Temperature distribution of TiNi SMA subjected to stress induced phase transformation during loading (martensite transition). The numbers above the thermograms correspond to the proper points of the stress-strain curve shown in Fig. 2

(Fig. 4; thermogram 9). In this way, during the SMA loading, the temperature distribution accompanying the process of origin and progress of the martensitic transformation was inhomogeneous — the increase of temperature in some areas of the specimen became much higher than in another. It was related to the phase transformation process that is strongly temperature dependent.

When the strain increased, a still higher and higher number of lines (bands) of the new material phase, characterised by a significantly higher temperature, were registered (Figs. 4, 5). Nevertheless, it was observed for all the tests, that the phase transition started in the central area of the specimen and developed in both directions towards

the specimen grips (Figs. 4, 5). During the stress-strain "plateau", when the martensitic transition started, the thermograms No. 4–9 (Fig. 4) were registered. Next, the thermograms No. 10–12 (Fig. 5), related to the slightly increasing segment of the stress-strain curve, were obtained. After this stage, just when the bands of the new phase reached the specimen grips (Fig. 5; thermogram 12), the angle of inclination of the stress-strain curve started to be slightly higher (Fig. 2). The highest temperature, probably related to the still developing phase transformation, was observed and registered in the central area of the specimen. The temperature change registered at the point where the phase transition start

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was noticed  $(\Delta T_P)$ , was higher than the average temperature  $(\Delta T)$  taken from the chosen specimen area (look at the temperature vs. strain curves presented in Fig. 2).

At higher strain, the regions of higher temperature became less clearly defined, most likely because of the further martensite-phase development in the whole material volume and the heat flow to the surroundings. When the martensitic transformation was almost completed, the temperature of the specimen surface became more homogeneous again (Fig. 6; thermogram No. 14). Furthermore, during the following tests carried out on the same TiNi SMA material, it turned out that the specimen could be loading further, in wider strain range, without entering the plastic range of the new, martensite phase. It means that the "safe" SMA deformation range could be wider.

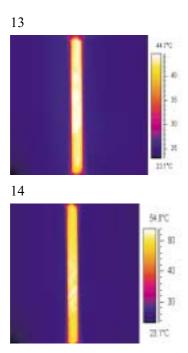


Fig. 6. Temperature distribution of TiNi SMA subjected to stress induced phase transformation during loading (martensite transition). The martensitic transition is almost completed. The numbers shown above the thermograms correspond to the proper points of the stress-strain curve shown in Fig. 2

**5.2.** Reverse transformation. The heterogeneous field of the temperature distribution was also observed during unloading of the TiNi SMA, when the reverse transformation, accompanied by significant temperature decrease, took place (Figs. 2, 7).

When unloading the specimen the temperature drops as the stress decreases. First, at the elastic unloading stage a slow temperature drop can be observed probably due to heat exchange with the surroundings. Then, the temperature drops rapidly due to the endothermic reverse transformation. At the last stage a drop in the mean specimen temperature slows down remaining at almost the same level until the unloading process ends.

At the beginning of the unloading, the temperature distribution was almost homogeneous — there were not too many differences on the specimen surface. After the initial stage of the reverse transformation, still almost uniform, the temperature distribution became inhomogeneous. On the all specimen surface the narrow bands of lower temperature in two almost perpendicular directions were observed. The bands were related to appearance of the parent, austenite phase. As the stress and strain decreased, the bands were characterised by the higher temperature decrements. During the more advanced process of unloading, at the end of reverse transformation, the registered narrow bands of various colors, related to the different temperature, were almost uniformly distributed on the all specimen surface area (Fig. 7; thermogram 20).

After the unloading to null, when the reverse transformation (martensite into austenite) was almost completed, the temperature of the specimen falls down with the drop up to 5 K, even beyond the initial temperature in some area. The lowest temperature was registered at the cross-section of the interphase bands.

The temperature distributions registered after complete unloading of the specimen were shown in Fig. 8. After 0.98 s of the specimen unloading: Fig. 8; thermogram 21a, the temperature distribution was still heterogeneous. Even after 3.20 s of the unloading to null: Fig. 8; thermogram 21b, the temperature distribution still remained non-uniform.

#### 6. Discussion

The investigations conducted have proved that during the phase transitions the stress magnitudes increase as the temperature at which the deformation process develops increases. The average temperature changes  $(\Delta T)$ , as a function of stress in TiNi SMA subjected to stressinduced phase transformations were presented in Fig. 9. Presenting mean sample temperature changes as a function of stress makes the analysis of phase transformations more clear. The maximum increase in temperature related to the martensitic transformation, was quite significant, up to 30 K in some area: the higher temperature was measured at the cross-section of the interphase bands. When unloading the specimen the temperature drops as the stress decreases. First, at the elastic unloading stage a slow temperature drop can be observed probably due to heat exchange with the surroundings. Then, the temperature drops rapidly due to the endothermic reverse transformation. During the unloading, when the reverse transformation (martensite into austenite) took place, the temperature of the specimen falls down, in some area with the drop up to 5 K below the initial temperature: the lower temperature was measured at the cross-section of the interphase bands (Fig. 7).

Particularly interesting are also the results obtained after unloading of the strain-controlled test. Namely, after

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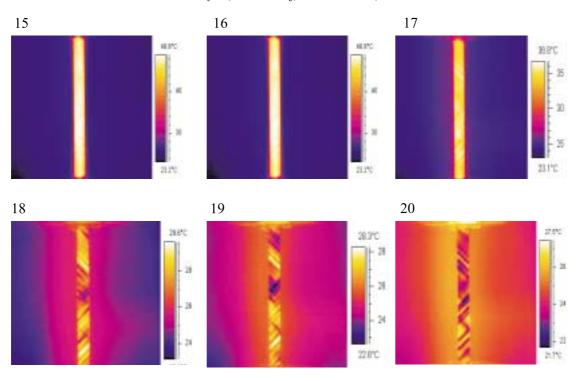


Fig. 7. Temperature distribution of TiNi SMA subjected to stress induced phase transformation during unloading (reverse transition).

The numbers above the thermograms correspond to the proper points of the stress-strain curve shown in Fig. 2

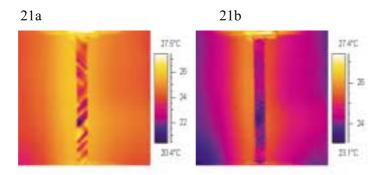


Fig. 8. Temperature distribution of TiNi SMA subjected to stress induced phase transformation after unloading: The numbers above the thermograms correspond to the point 21 of the stress-strain curve shown in Fig. 2; 21a - 0.98 s after the specimen unloading, 21b - 3.20 s after the specimen unloading

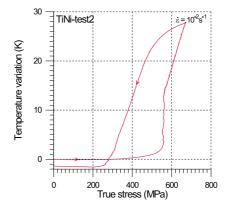


Fig. 9. Temperature changes vs. stress in TiNi SMA subjected to stress induced phase transformations

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3.20 s of the unloading to null, the temperature distribution remained still heterogeneous (Fig. 8). It could be explained like this. After unloading, some residual martensite remained. That is, though the reverse transformation completed macroscopically, it did not microscopically complete. Based on this residual martensite, a residual or internal stresses after the unloading might appear. Therefore, some local microscopic reverse transformation still occurred in the elastic region of unloading after the macroscopic completion of the reverse transformation. Due to this effect, the heterogeneous temperature distribution may appear after unloading. It would be cleared up in research concerning cycling tests of the TiNi SMA.

#### 7. Conclusions

Basing on the results obtained from tests on specimens made of the TiNi Shape memory alloy subjected to stress induced phase transformation in terms of mechanical characteristics and temperature changes, the following concluding remarks were proposed.

The thermomechanical behaviour of TiNi SMA confirmed the exothermic character of the austenite into martensite transformation and the endothermic character of the reverse one: the average temperature changes were up to 30K for the test carried out with this strain rate.

The temperature distributions registered during loading and unloading of the TiNi SMA were inhomogeneous in the ranges of martensite and reverse transformations which demonstrate that both the transformations occurred in inhomogeneous way, via subsequent bands of new phase.

The narrow bands of the significantly higher temperature related to the martensitic phase were observed to start from the central part of the specimen and developed towards the specimen grips; they were characterized by temperature increase about 6-10 K; their angle of inclination was about 42 degree. For higher stresses, the lines of new phase were also developed in the perpendicular direction; the highest temperature was registered at the cross-section of the bands.

At higher strain, the regions of higher temperature became less clearly defined, most likely because of the further martensite phase development in the whole material volume and the heat flow to the surrounding. When the martensitic transformation was almost completed, the temperature of the specimen surface became almost homogeneous again.

The lines of reverse transformation observed during the unloading started and developed in whole the material volume, however the process was still inhomogeneous, the bands of new phase were wider in comparison to the martensitic fronts and were characterized by the temperature decrease of about 6–10 K; their slope of inclination was also about 42 degree.

During the more advanced process of unloading, at the

end of reverse transformation, the registered narrow bands of various colors, related to the different temperature, were almost uniformly distributed on the all specimen surface area.

After complete unloading of the specimen the temperature distribution was still inhomogeneous which was probably related to the residual martensite, still remained in the material of the specimen.

The infrared camera turned out to be really good tool for investigation of the kinetics of stress induced phase transformations, namely the origin and further evolution of new phase in TiNi shape memory alloy.

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#### References

- [1] J. A. Shaw and S. Kyriakides, "On the nucleation and propagation of phase transformation fronts in a TiNi Alloy", *Acta Mater.* 45, 683–700 (1997).
- [2] John A. Shaw, "Simulation of localized thermo-mechanical behaviour in NiTi shape memory alloy", Plasticity~16,~541-562~(2000).
- [3] H. Tobushi, K. Takata, Y. Shimeno, W. K. Nowacki and S. P. Gadaj, "Influence of strain rate on superelastic behaviour of TiNi shape memory alloy", *Proc. Instn. Mech. Engrs.*, 213(L), 93–102 (1999).
- [4] Pingua Lin, Hisaaki Tobushi, Akira Ikai and Kikuaki Tanaka, "Deformation properties associated with the martensitic and R-phase transformations in TiNi Shape Memory Alloy", J. Appl. Biomechanics 10, 1–11 (1995).
- [5] S. P. Gadaj, W. K. Nowacki and H. Tobushi, "Temperature evolution during tensile test of TiNi shape memory alloy", Arch. Mech. 51, 649–663 (1999).
- [6] S. P. Gadaj, W. K. Nowacki and E. A. Pieczyska, "Temperature evolution in deformed shape memory alloy", *Infrared Physics & Tech* 43, 151–155 (2002).
- [7] D. Helm and P. Haupt, "Thermomechanical behaviour of shape memory alloys", *Proc. SPIE* 4333, 302–313 (2001).
- [8] E. A. Pieczyska, S. P. Gadaj and W. K. Nowacki, "Thermoelastic and thermoplastic effects in steel, polyamide and shape memory alloys", Proc. SPIE 4710, 479–487 (2002).
- [9] E. A. Pieczyska, S. P. Gadaj, W. K. Nowacki and H. Tobushi, "Investigations of nucleation and propagation of phase transitions in TiNi SMA", *QIRT Journal* 1, 117–128 (2004).
- [10] K. Tanaka, S. Kobayashi and Y. Sato, "Thermomechanics of transformation pseudoelasticity and shape memory effect in alloys", *Inter. J. Plasticity* 2, 59–72 (1986).
- [11] K. Tanaka, "A Thermomechanical sketch of shape memory effect: one-dimensial tensile behaviour", Res Mechanica 18, 251–263 (1986).
- [12] H. Tobushi, K. Okumara, M. Endo and K. Tanaka, "Deformation behaviour of TiNi shape memory alloy under strain- or stress-controlled conditions", *Arch. Mech.*, 54 75–91 (2002).