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TEMPERATURE MEASUREMENT AS A NEW TECHNIQUE APPLIED TO THE PHASE TRANSFORMATION STUDY IN A TiNi SHAPE MEMORY ALLOY SUBJECTED TO TENSION

POMIARY TEMPERATURY PODCZAS ODKSZTAŁCANIA STOPU TiNi Z PAMIĘCIĄ KSZTAŁTU JAKO METODA BADANIA PRZEMIAN FAZOWYCH

Temperature distributions obtained from studying the stress induced phase transformations in a TiNi shape memory alloy (SMA) were employed for the investigation into nucleation and further development of the bands of martensite and reverse transformations. A thermovision camera was used to register the distribution of infrared radiation emitted by the specimen surface and constructed thermograms with 50 Hz frequency. Basing on temperature changes and the relevant mechanical characteristics it was noticed that just after crossing a certain threshold stress, narrow lines of considerably higher temperature — up to 10 K, corresponding to the martensite phase, appeared starting from the central part of the specimen and developing towards the specimen grips. Their angle of inclination was about 42°. At higher stresses, a few such lines parallel to each other occurred and moved towards the specimen borders, as well as the next "family" of them, developing in the perpendicular direction. The heterogeneous field of the temperature distribution was observed also during the unloading process of TiNi SMA, while the reverse transformation — austenite into martensite, took place. The reverse transformation was accompanied by a significant temperature decrease. The lines of reverse transformation were observed when the unloading process started and developed in the whole material volume, however the process remained inhomogeneous. The processes of relaxation do not bring about any changes in the nature of phase transitions, however, the relaxation involved temperature changes cause stress changes as the deformation (unloading) develops after relaxation.

Pomiary rozkładów temperatury podczas przemiany martenzytycznej i przemiany odwrotnej stopu TiNi z pamięcią kształtu, stymulowanych odkształceniem, wykorzystano do badania inicjacji i rozwoju tych przemian oraz wpływu parametrów odkształcania na te przemiany. Rozkłady temperaury mierzono za pomocą kamery termowizyjnej, umożliwiającej

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uzyskiwanie obrazów termicznych z częstotliwością do 50 Hz. Na podstawie badań charakterystyk mechanicznych i zmian temperatury stwierdzono, że mertenzytyczna przemiana fazowa rozpoczyna się pod kątem ok. 42° do kierunku rozciągania próbki. W miarę odkształcania pojawia się coraz więcej pasm, zarówno równoległych, jak i skierowanych przeciwnie, aż obejmują one całą próbkę. Skok temperatury w miejscu pojawienia się pojedynczego pasma wynosi ok. 6 K. Przemiana odwrotna jest również procesem niejednorodnym, przy czym w pasmach tej przemiany następuje spadek temperatury. Przyrost średniej temperatury próbki podczas przemiany martenzytycznej może przekraczać 40 K, co powoduje przyrost naprężeń w miarę rozciągania. Procesy relaksacji nie powodują zmian charakteru przemian fazowych, jednak związane z relaksacją zmiany temperatury wywołują zmiany naprężeń przy kontynuacji odkształcania (odciążania) po relaksacji.

1. Introduction

The properties of shape memory materials are highly temperature dependent. The phenomenon of shape memory is associated with the reversible martensite transformation of the following characteristic temperatures: M_s (martensite start), M_f (martensite finish), A_s (austenite start), A_f (austenite finish) and M_d (martensite dead) — the values of which depend on the type of material considered.

Phase transitions in shape memory materials may be deformation induced and in that case the stress-strain material characteristics depend on the temperature at which the material starts to deform (see Fig. 1). If the material undergoes deformation at a temperature below A_f the stress induced transformation of the primary phase (austenite) is observed and after being unloaded the specimen remains deformed. After heating the specimen above A_f the deformation disappears (Fig. 1a) due to the so-called shape memory effect. The deformation process undergoing at a temperature slightly above A_f involves the reversible martensite transformation, i.e. the phenomenon of pseudo-elasticity (Fig. 1b). On the other hand, if the deformation develops at a temperature much higher then the transformation temperature (M_d) the stress-strain curve reveals a shape typical for the austenitic structure (Fig. 1c).

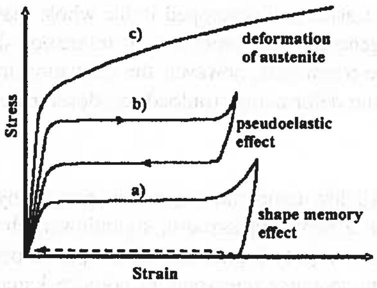

Fig. 1. Typical stress-strain curves of TiNi SMA at different temperature: a) shape memory effect ($T < A_f$), b) phenomenon of pseudo-elasticity ($T > A_f$), c) shape typical for the austenitic structure

The characteristics shown in Fig. 1 illustrate the process of TiNi SMA deformation developing at very low strain rates ($\dot{\varepsilon} < 10^{-4}$). The magnitudes of stress as well as the shape of stress-strain curves depend crucially on the strain rate and temperature of the deformation process.

The investigations conducted have proved [1 ÷ 6] that during the phase transitions the stress magnitudes increase as the strain rate grows and as the temperature at which the deformation process develops increases.

At the strain rates used in practice the stress induced phase transitions are associated with temperature changes in the examined body, which, in turn, affect the mechanical characteristics; i.e. involve the stress increase, that influences the process of phase transition.

The investigations into temperature changes accompanying the deformation process of shape memory materials have been launched quite recently. At first the temperature changes were measured using a thermocouple [7], which, however, limited the monitoring of changes to a single chosen point only. Application of infrared cameras to monitoring and registration of temperature changes over chosen areas of the examined specimen facilitated a substantial progress. Investigations of this type allow for observing of initiation and development of phase transition fronts [3, 8, 9], as well as for measurement of a mean [3, 5, 10] temperature, together with a temperature at an arbitrary chosen point on the specimen surface for a variety of parameters and kinds of deformation and at different measurement temperatures, respectively.

The results of investigations into mechanical characteristics and temperature distributions in the TiNi SMA with the characteristic temperature $A_f \sim 288$ K subjected to tension have been presented in the paper. The phenomenon of superelasticity can be observed in that alloy at ambient temperature; i.e., after the stress induced martensite transformation ends, during the unloading the complete reverse transformation develops spontaneously. The measurements of mechanical characteristics and temperature distributions allowed the influence of different factors upon the initiation and development of the stress induced martensite and reverse transformations, respectively, to be studied in that alloy.

2. Material characteristics and measurement technique

The tests of TiNi alloy of the constitution: 55,3wt% Ti and 44.7wt% Ni were made on the specimens of dimensions 160 mm × 10 mm × 0,4 mm ($A_f \sim 288$ K), cut off a strip of the same cross section. The measurements were made at an ambient temperature of about 295K. The specimens were subjected to tension using a testing machine of hydraulic drive. In the course of investigations both the mechanical characteristics and intensity distribution of the infrared radiation emitted by the specimen surface were registered. Those distributions were registered using infrared equipment allowing for the infrared photographs, i.e. thermograms, to be stored in a digital form with a frequency of 50Hz. That type of storing allows for reproduction of these images at any moment, and makes the calculations of temperature straightforward (as well as the presentation) as a function of time or other parameters of the deformation process.

A mean temperature of the specimen was calculated over an area of dimension 8 mm × 60 mm located in the central part of the specimen. To make its emissivity higher and more homogeneous the specimen surface was covered with a very thin layer of carbon black powder.

A typical stress-strain characteristic of TiNi alloy subjected to tension obtained at ambient temperature is shown in Fig. 2. After an initial stage of deformation the martensite transformation starts. After it ends the material (at the martensite phase) starts to deform in an elastic form.

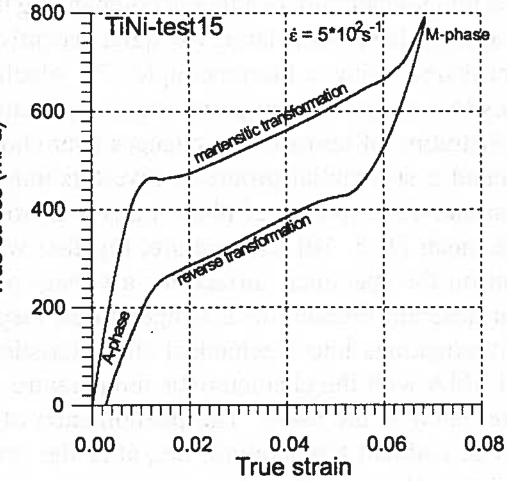

Fig. 2. Stress-strain characteristic of TiNi SMA strained at ambient temperature

During the process of unloading, after passing its elastic stage the reverse transformation initiates. After it ends the material almost completely returns to the parent austenite phase.

Typical curves illustrating the phenomenon of pseudo-elasticity in SMAs, obtained at very low strain rates [1, 7], have shown that the segments of $\sigma(\varepsilon)$ diagrams corresponding to the martensite and reverse transformations, respectively, are nearly parallel to the axis x. In deformations occurring within the range of quasi-static strain rates some heat is released during the martensite transformation that involves an increase in the specimen temperature reaching 40 K (see Section 4). Changes in the material temperature, in turn, affect a stress increase in the specimen during the martensite transformation [11].

Therefore, the angle of inclination of the curve $\sigma(\varepsilon)$ changes. Similar phenomena occurring, however, in the opposite direction, can be observed during the unloading process when the reverse transformation takes place.

3. Initiation and development of the phase transition fronts

Phase transitions are always accompanied by the heat emission or absorption, i.e. are associated with temperature changes. The analysis of temperature changes on the specimen surface allows for examination of initiation and development of the transition fronts. A high frequency of the thermograms applied (up to 50Hz) enables a relatively thorough analysis of the aforementioned phenomena within the considered range of strain rates: $10^{-3}\ \mathrm{s}^{-1} \div 10^{-1}\ \mathrm{s}^{-1}$.

For analysis purposes the mechanical characteristics obtained in deformation were combined with the thermograms illustrating the phase transitions — a specimen operation is illustrated in Fig. 3 and Fig. 4. One can see that the points corresponding to the thermograms shown in Fig. 4 are marked on the stress-strain curve (Fig. 3) and the numbers over thermograms correspond to the points marked on the curve. The thermograms were chosen that were very characteristic of the phenomena occurring during the martensite and the reverse transitions.

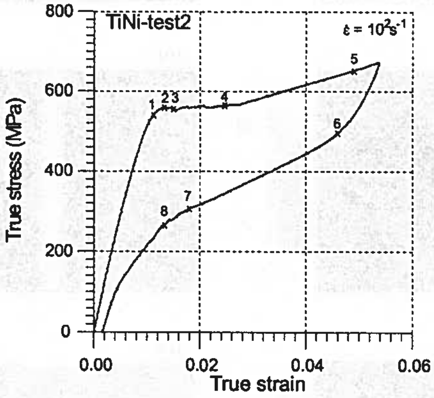

Fig. 3. Stress-strain curve of TiNi SMA obtained at the constant rate of deformation ($10^{-2}\ \mathrm{s}^{-1}$); marked points correspond to thermograms shown in Fig. 4

Before the tension start the specimen 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 grows, the thermal image, however, remains almost quite homogeneous indicating homogeneous nature of the deformation process (see Fig. 4(1)).

When the true strain value reaches $\varepsilon = 0.013$, i.e. the value corresponding to the local maximum stress, a line evolving then into a narrow band of higher temperature appears on the specimen surface proving the initiation of martensite transformation (Fig. 4(2)). The line makes the angle of 42° with the direction of tension. The temperature difference in the area where the band appears is equal to 6 K, proving rapid nature of the process. As the tension proceeds the band significantly widens and other lines appear at first parallel and then inclined at the same angle but in

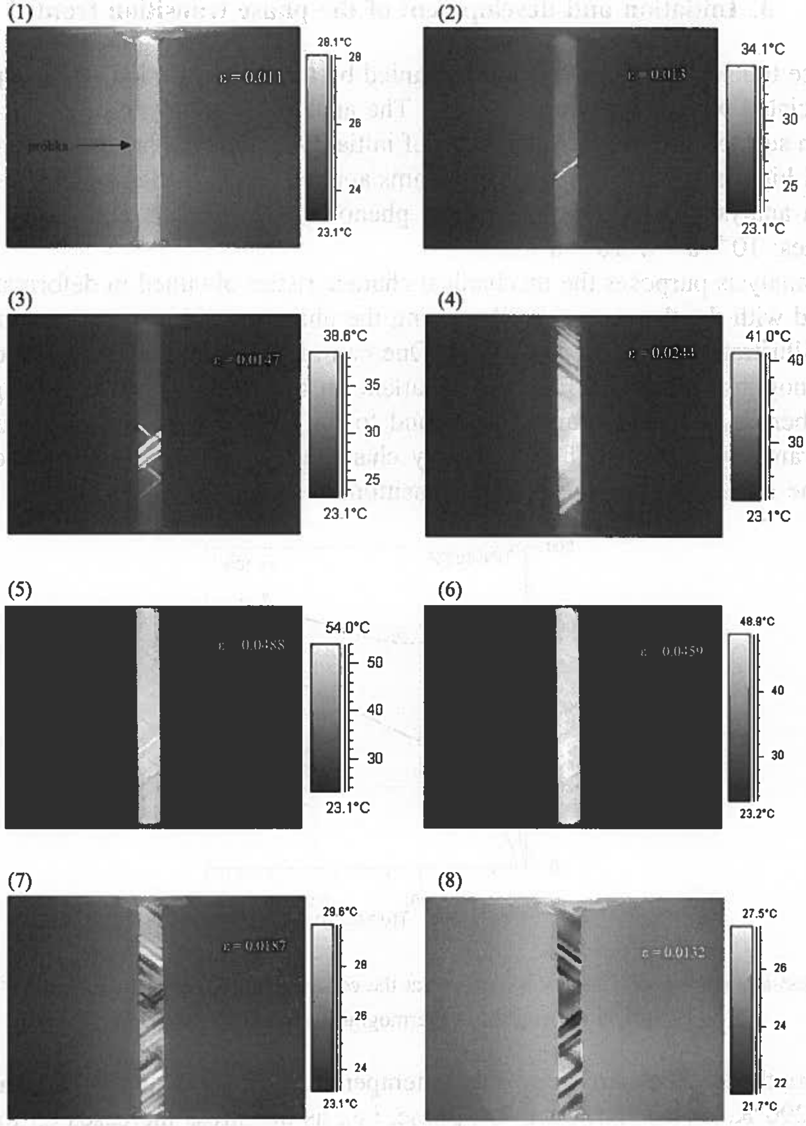

Fig. 4. Temperature distribution of TiNi SMA subjected to uniaxial tensile test at room temperature with constant strain rate of deformation (10^{-2} s^{-1}); the numbers of thermograms correspond to points at the curve in the Fig. 3

the opposite direction (Fig. 4(3)). At higher strains more and more lines evolving into bands appear (Fig. 4(4)). Due to a heat flow the thermal image becomes more ambiguous and the mean temperature of the specimen surface increases. At the ending phase of tension the temperature increase in the bands of transformation exceeds 30 K (Fig. 4(5)).

In the course of unloading, after passing the elastic phase, the thermal image of the specimen (Fig. 4(6)) is very similar to that obtained at the tension end, despite the specimen temperature drop due to heat exchange with the surroundings.

As the unloading proceeds, the bands of lower temperature appear, proving that the reverse transformation develops also in an inhomogeneous way (Fig. 4(7)). Biggest temperature drops can be observed in the bands located on the specimen surface, up to 10 K. However, the temperature these areas at the unloading end (Fig. 4(8)) is only a little bit lower than the initial temperature of the specimen (before testing).

From the investigations conducted it can be concluded that the stress induced martensite transformation in TiNi SMA is an inhomogeneous process. It initiates and develops in transformation bands similar to the Lüders bands. Also the reverse transformation is inhomogeneous. Since the martensite transformation in TiNi SMA is accompanied by heat emission it is an exothermic process while the reverse transformation is the endothermic one.

4. Mean temperature changes

After analysis of changes in the mean temperature of the specimen surface one can draw suggestions concerning the courses of effects accompanying the deformation induced phase transitions 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.

Figure 5 shows the diagrams of mean temperature changes and stresses as a function of deformation obtained in the courses of loading and unloading, respectively, of the specimen within the range of phase transitions with the strain rate $\dot{\varepsilon} = 10^{-2}$ s^{-1} (corresponding to the thermograms shown in Fig. 4). The figure presents also the temperature changes at a point located in the central area of the band of phase transitions (denoted by ΔTp).

The mean temperature of the specimen (ΔT — Fig. 5) increases after passing through the elastic phase of deformation. However, at the stage at which the bands of martensite transformation have developed over the whole specimen length (see Fig. 4(4)) the growth slightly slows down which corresponds to a slight inflexion visible on the curve $\sigma(\varepsilon)$ (Fig. 5). Then the temperature increases until the phase transition ends.

In the course of unloading the mean temperature drops until the curve $\sigma(\varepsilon)$ reaches its inflexion point, which corresponds to the deformation at which the bands of martensite transformation appear during tension. At the ending phase of unloading (the elastic unloading phase — see Fig. 5) the mean temperature changes are unobservable.

The temperature measured at a point located within the area where the first line of martensite transformation (ΔTp — Fig. 5) has appeared on the specimen surface, increases starting from the tension beginning (like the mean temperature) since the specimen material remains at the homogeneous deformation phase. At the moment

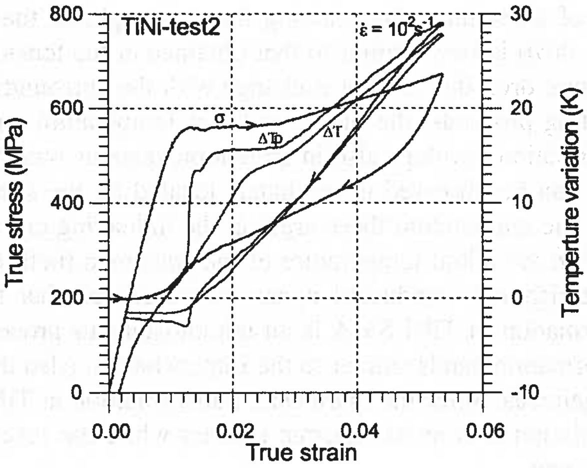

Fig. 5. Stress-strain and temperature changes curves of TiNi SMA strained with the rate of deformation equal 10^{-2} s^{-1}: ΔT — average temperature change from the specimen area and ΔTp — temperature change in the point where the phase transition start was noticed

when the transformation initiates a rapid temperature growth of about 6 K is observed. Then, as the tension proceeds, the temperature changes are affected by the specimen translation relative to the point selected on the thermogram, which is immovable relative to the point selected on the thermogram. When the point appears outside the transformation band its temperature does not increase being equal to that of the surrounding areas. After the homogeneous deformation connected with the development of transformation bands ends the temperature at the chosen point changes in a way similar to the mean temperature changes, revealing however higher values.

In the course of unloading the temperature changes at the chosen point are almost identical to those of the mean temperature until the deformation stage is reached at which the point is located in the area of the reverse transformation. A rapid temperature drop of about 4 K appears at that moment followed by a small growth due to a heat flow from the neighboring areas (Fig. 5).

During the tension test performed at higher strain rate ($\dot{\varepsilon} = 10^{-1}$ s^{-1}) changes in the mean temperature observed during martensite transformation reveal a similar nature (see Fig. 6). The inflexion points can be also seen in the diagrams of stress and mean temperature, respectively, appearing in the area where the bands of martensite transformation occur over the whole specimen length. The increase in the mean temperature after the transformation process ends reaches almost 40 K.

The course of temperature changes in unloading is similar to that observed at a lower strain rate. The temperature drop, however, is smaller, probably due to the thermal inertia observed at a higher strain rate.

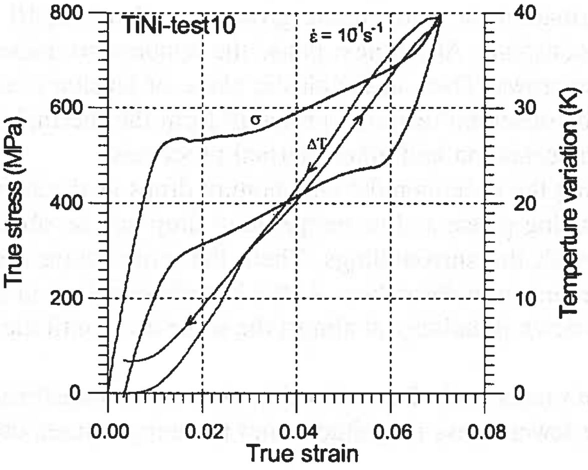

Fig. 6. Stress-strain and mean temperature changes curves of TiNi SMA at the rate of deformation equal 10^{-1} s^{-1}

Presenting mean specimen temperature changes as a function of stress makes the analysis of phase transformations more clear. Figure 7 presents those diagrams for specimens subjected to tension with the different strain rates; i.e., $5{\times}10^{-3}$ s^{-1}, 10^{-2} s^{-1} and 10^{-1} s^{-1}.

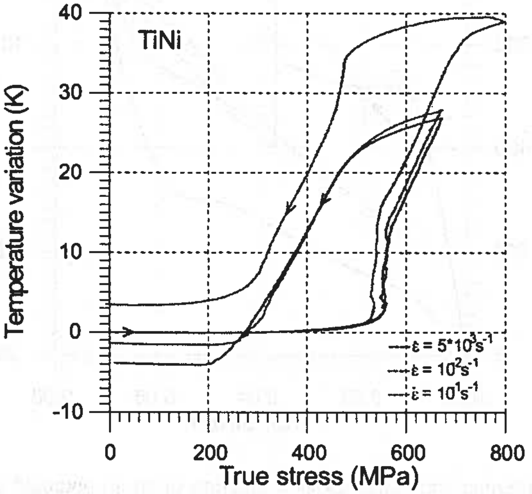

Fig. 7. Stress dependence of mean temperature change for various strain rates of TiNi SMA

One can observe that the courses of diagrams presented are of similar nature. At the elastic phase up to the stress of about 400 MPa no changes are observed in the mean temperature of the specimen. At the subsequent phase the temperature increases slowly not exceeding the values of 2 ÷ 3 K. Over the whole phase of inhomogeneous martensite transformation the temperature growth equals about 10 K (with the stress remaining almost constant). At the next phase the temperature increases slightly more slowly as the stress grows. Then, at the elastic phase of tension a rather small temperature growth can be observed resulting probably from the thermal inertia during both the martensite transformation and other thermal processes.

When unloading the specimen the temperature drops as the stress decreases. First, at the elastic unloading phase 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 phase a drop in the mean specimen temperature slows down remaining at almost the same level until the unloading process ends.

At higher strain rates both the martensite and reverse transformations initiate and develop at slightly lower stress magnitudes and the temperatures observed are higher.

5. Relaxation influence on the phase transformation phenomenon

The investigations conducted into relaxation, i.e. stress decay during forced deformation of the TiNi SMA followed the scheme:

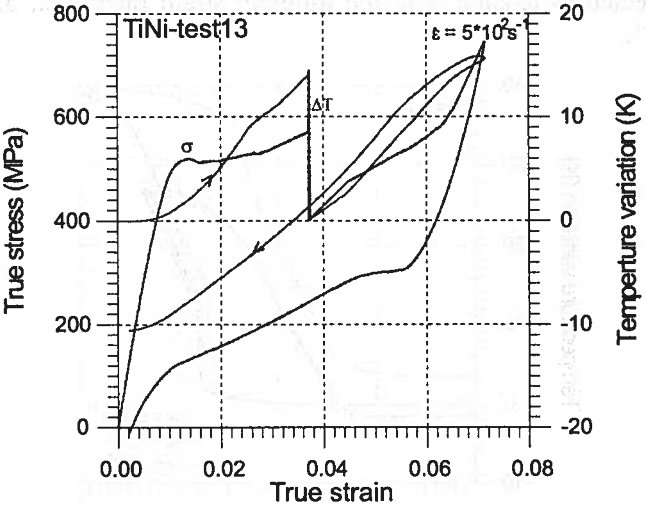

Fig. 8. Stress and mean temperature changes as a function of strain obtained during straining with the relaxation in the branch of loading

- tension at a constant strain rate ($\dot{\varepsilon} = 5 \times 10^{-2}s^{-1}$) until a given strain value is reached,
- maintaining the strain value at the same level for 3 minutes,
- subsequent loading and/or unloading until a typical cycle of phase transitions ends.

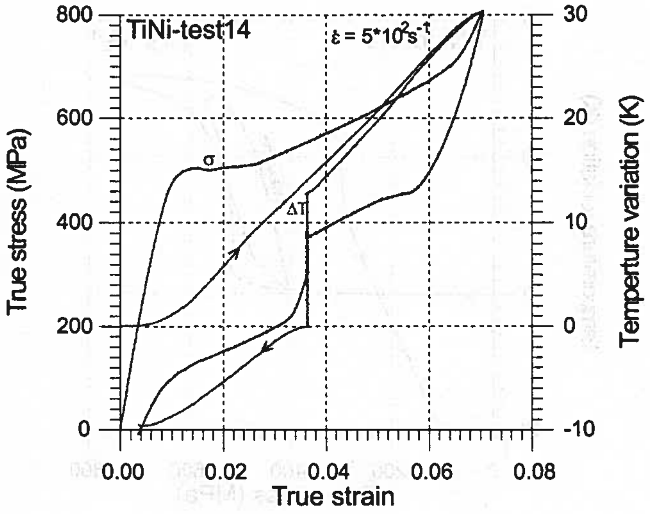

Fig. 9. Stress and mean temperature changes as a function of strain obtained during straining with the relaxation in the branch of unloading

The following two types of test were performed:

— with the relaxation stage in the course of loading (Fig. 8),

— with the relaxation stage in the course of unloading (Fig. 9).

Figures 8 and 9 show the changes in stress and mean temperature of the specimen surface, respectively, as a function of deformation that have resulted from the aforementioned tests.

Before relaxation the courses of stress and temperature changes reveal a typical form observed in such tests. During the relaxation phase in both the above described cases a monotonic stress drop is observed, with smaller values observed along the unloading branch of the curve, while the temperature drops monotonically to the initial temperature of the specimen. Then in the case of relaxation in the loading branch (Fig. 9), a temperature growth of about 15 K is observed followed by a drop in unloading to about 10 K, below the initial temperature of the specimen. On the other hand, after relaxation in the unloading branch further temperature drop is observed to about 10 K below the initial temperature of the specimen.

The diagrams of mutual relations between changes in the mean specimen temperature as a function of stress (Fig. 10 and Fig. 11) are very interesting as well.

Before relaxation those changes are typical of such processes (see Fig. 7). At the relaxation phase appearing in the loading branch (Fig. 10) the temperature increases slightly at the initial stage of stress drop and then fall. The temperature courses versus the stress in loading and unloading, respectively, prove that the processes of phase transitions develop initiating, however, at a considerably lower stress level. In the case of relaxation appearing in the unloading branch (Fig. 11) at first the temperature drop slows down. After relaxation the reverse transformation develops.

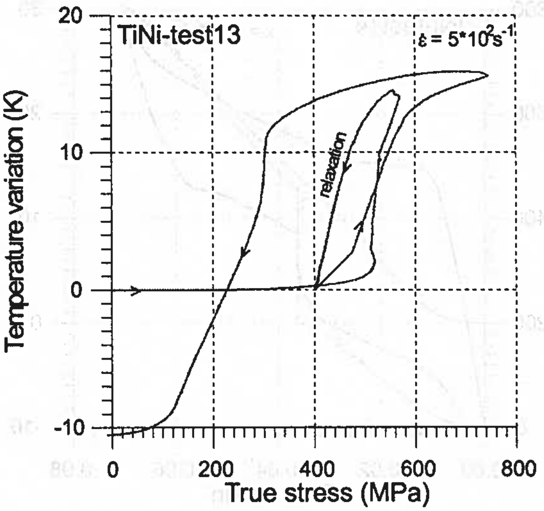

Fig. 10. Stress dependence of mean temperature change obtained during straining of the specimen with the relaxation in the branch of loading

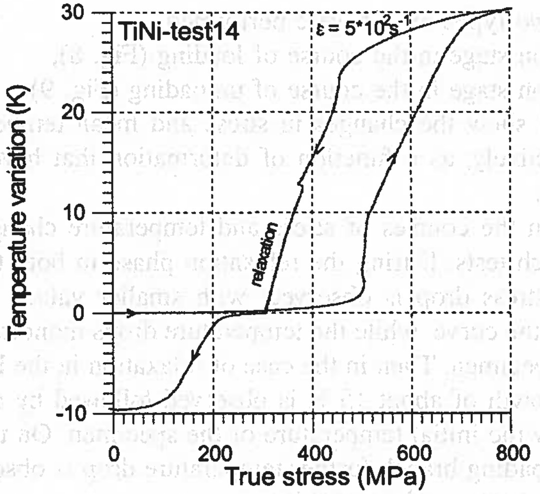

Fig. 11. Stress dependence of mean temperature change obtained during straining of the specimen with the relaxation in the branch of unloading

6. Final remarks

Basing on the results obtained from tests on specimens made of the TiNi SMA subjected to tension in terms of mechanical characteristics and temperature changes the process of deformation induced martensite transformation can be divided into the following stages:

1) Homogeneous deformation comprising the ranges of elastic deformation and the so-called R-phase; revealing a homogeneous and relatively small temperature increase
2) Inhomogeneous martensite transformation, while the narrow bands of phase transition initiate and develop; with a rapid mean temperature growth of about 10 K appearing at a very small stress increase
3) Development of moderately homogeneous phase transition; revealing sharp temperature growth as the stress increases; rise in the strain rate at this phase causes a temperature growth, which in turn involves a stress increase since at higher temperatures the phase transition initiates at higher stresses
4) Elastic tension of the martensite phase after the phase transition ends.

During unloading the following phases can be distinguished:

1) Elastic unloading with a small temperature drop, appearing mainly due to heat exchange with the surroundings
2) Homogeneous reverse transformation; revealing a substantial drop in the mean temperature down to the value close to the initial temperature of the specimen (depending on the strain rate applied)
3) Rapid temperature drop in the bands of reverse transformation that appear, mean temperature reaching the value of 10 K below the initial temperature of the specimen
4) Elastic unloading, when the temperature changes depend on both the heat exchange with the surroundings and thermo-elastic effects occurring in the material of the specimen.

The relaxation processes appearing in both the loading and unloading branches of deformation do not exert a crucial influence on the nature of phase transitions. The results obtained indicate that after relaxation in both the loading and unloading branches the phase transition processes develop, initiating however at substantially lower stresses and temperatures of the specimen.

Acknowledgements

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