

E. PIECZYSKA*, J. DUTKIEWICZ**, F. MASDEU***, J. LUCKNER*, R. MACIAK*

INVESTIGATION OF THERMOMECHANICAL PROPERTIES OF FERROMAGNETIC NiFeGa SHAPE MEMORY ALLOY SUBJECTED TO PSEUDOELASTIC COMPRESSION TEST

BADANIE WŁAŚCIWOŚCI TERMOMECHANICZNYCH FERROMAGNETYCZNEGO STOPU Z PAMIĘCIĄ KSZTAŁTU NiFeGa W PROCESIE PSEUDOSPŁĘZYSTEGO ŚCISKANIA

In the study stress-induced reversible phase transformation in NiFeGa magnetically controlled shape memory alloy subjected to pseudoelastic compression test was investigated. The specimen's mechanical characteristics and temperature changes related to the exothermic martensite transformation and endothermic reverse transformation were measured in contact-less way by using a fast and sensitive infrared camera (IR). It was found that the stress-induced phase transformation process occurs in this alloy in heterogeneous way, since the observed specimen's temperature distribution was not uniform. Stress-strain curves obtained for the first, as well as for the subsequent six loading-unloading compression cycles and their related temperature changes, elaborated as average from the specimen's surface, were analyzed. It was concluded that the stress and the temperature changes developing in the subsequent cycles depend on the applied test conditions, however the highest discrepancies were observed between the first and the second cycles of the compression loading.

Keywords: magnetically controlled shape memory alloys, localized phase transformation, stress-induced transformation, compression test, temperature measurement, infrared camera

W pracy przedstawiono wyniki badań zmian parametrów mechanicznych oraz temperatury stopu Ni₅₄Fe₁₉Ga₂₇, wykazującego magnetyczną pamięć kształtu. Próbkę stopu poddawano procesowi pseudosprężystego ściskania. Temperaturę mierzono za pomocą szybkiej kamery termowizyjnej. Stwierdzono, że indukowana naprężeniem przemiana fazowa zachodzi w tym stopie w sposób niejednorodny, a przebieg charakterystyk mechanicznych i zmian temperatury w kolejnych cyklach obciążania i odciążania próbki zależy od zastosowanej metodyki badawczej.

1. Introduction

Ferromagnetic shape memory alloys (FSMA) are novel multifunctional materials, discovered by Ullakko in 1996 [1] which exhibit magnetic field induced reversible strains of up to 10%. Similarly to classic shape memory alloys (SMA), these alloys in most cases have a high-symmetry cubic structure in the high-temperature phase and a low-symmetry tetragonal structure in the low-temperature phase [2-5]. The low-temperature tetragonal structure is composed of regions with different crystallographic orientations, called the martensite variants. When the alloy is cooled to induce phase transformation, all variants are generated in almost equal amounts and with random arrangement, in order to minimize the change of the SMA speci-

men shape. Boundaries between the variants form twins which austenite-martensite interfaces can move easily. Meanwhile, when actuation of the alloys, based on the reorienting of the twin structure of martensite or the motion of the interfaces, is applied by stresses or by magnetic field, some orientations are privileged. Control of the magnetic field driven FSMA is rapid and can be realized with a high frequency of 50Hz-100Hz, while the frequency of acting of the classic SMA, e.g. TiNi, is only 1Hz.

Materials that develop rapid control exhibit a great potential in mechanical engineering. Piezoelectric and magnetostrictive materials exhibit rapid response, but their strokes are small. In classic shape memory alloys, strokes are large, but their control is rather slow, whereas control of the FSMA is fast and precise [3-5].

* INSTITUTE OF FUNDAMENTAL TECHNOLOGICAL RESEARCH PAS, 00-049 WARSZAWA, 21 ŚWIĘTOKRZYSKA STR., POLAND

** INSTITUTE OF METALLURGY AND MATERIALS SCIENCE PAS, 30-059 KRAKÓW, 25 REYMONTA STR., POLAND

*** UNIVERSITAT DE LES ILLES BALEARS, PALMA DE MALLORCA, SPAIN

Due to these advantages, the magnetically controlled shape memory alloys are applied as elements of sensors and actuators with demand of fast operation. Because of their cost, as well as a demand for the miniaturization of the control instruments, the sensors and the actuators often work in compression mode. The question is, how does the transformation occur in the FSMA subjected to compression, at which stresses it starts and develops, and how does the temperature accompanying the transformation process change?

In previous works, infrared imaging and thermo-mechanical parameters of the stress-induced exothermic martensite forward and endothermic reverse transformation were studied for classic TiNi SMA subjected to various kinds of deformation: tension [6-15], shear [14-16], torsion [17-19], torsion and fatigue [20]. Experimental and theoretical study of mechanical and thermal behavior of small disc specimens of composite, used in a car industry, subjected to compression, were carried out in [21], however it was not shape memory material.

In this paper, investigations of thermomechanical properties of the ferromagnetic $\text{Ni}_{54}\text{Fe}_{19}\text{Ga}_{27}$ polycrystalline subjected to subsequent pseudoelastic compression cycles of loading and unloading were carried out.

The goal of this study was to obtain answers for the following questions:

- Does the phase transformation process in the FSMA occur macroscopically homogeneously, or by nucleation and development of the new phase?
- Is it possible to record the temperature changes for so small specimens of the FSMA during the forward and reverse phase transformation in compression process?
- How does the alloy behave in subsequent cycles of loading and unloading?

The points raised above are crucial for the FSMA practical applications in new actuators.

2. Materials and experimental methods

The ferromagnetic shape memory alloy NiFeGa was cast by arc melting at Universitat de les Illes Balears, Palma de Mallorca. The composition in atomic percentage was as follows: $\text{Ni}_{54}\text{Fe}_{19}\text{Ga}_{27}$. It was FCC structure. Heat treatment at 1000°C during 6 hours was applied with low cooling to promote participation of γ phase in order to obtain the FSMA more ductile. The specimens were cut into small rectangular in the size of $9 \times 4 \times 4$ mm and their fundamental mechanical properties were obtained in Institute of Metallurgy and Materials Science, PAS, Cracow. Experimental investigation of the FSMA thermomechanical properties were carried out in the Institute of Fundamental Technological Research of PAS, Warsaw.

The all compression tests of FSMA were carried out with a strain rate equal to 10^{-2}s^{-1} . The mechanical characteristics were obtained by smart testing machine MTS. Two plates of mica were placed between the FSMA specimens and the compression grips of the testing machine in order to assure thermal isolation. The infrared radiation from the specimen surface was recorded in contact-less way by using a Flir System fast modern infrared camera, called Phoenix. The frame frequency of obtaining image was set to 538 Hz. The sensitivity of the temperature measurement was 0.025 K.

Two kinds of cycling compression tests were carried out for $\text{Ni}_{54}\text{Fe}_{19}\text{Ga}_{27}$ polycrystalline:

1. After each complete cycle of loading-unloading there was a pause until the temperature regained its initial level; denoted by KF12.
2. The subsequent cycles of loading- unloading were realized directly; denoted by KF11.

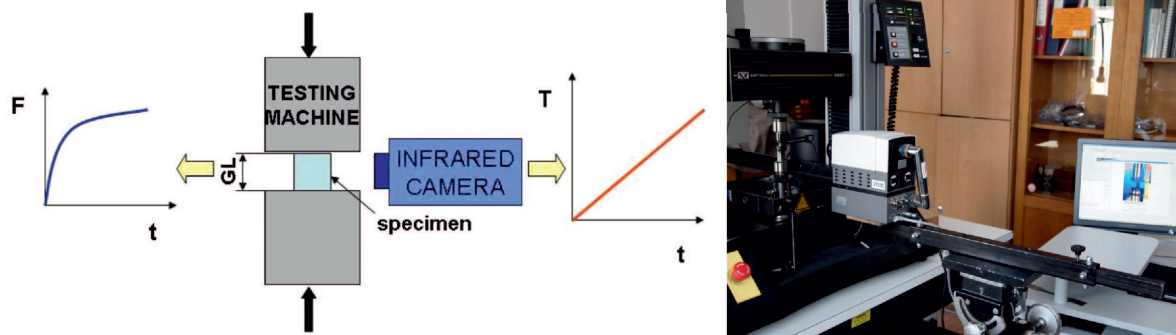


Fig. 1. Diagram and photograph of the experimental set up applied for investigation of mechanical and temperature characteristics of the FSMA loaded in compression test

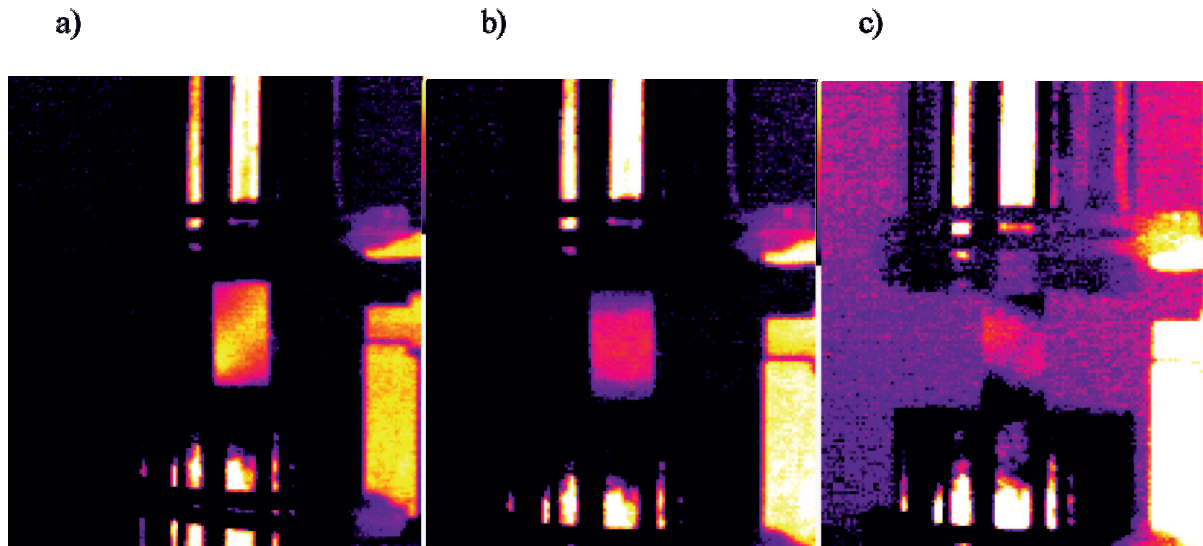


Fig. 2. Thermograms obtained by dynamic Phoenix infrared camera for the NiFeGa specimen subjected to compression: a) loading process, b) loading till strain limit, c) unloading process

On the grounds of the elaborated stress-strain curves and the related temperature changes the effects of thermomechanical couplings occurring during the stress-induced phase transformation in the FSMA subjected to subsequent loading-unloading cycles were studied.

3. Results and discussion

3.1. Monitoring of temperature distribution on the FSMA specimen surface during compression loading-unloading cycles

A thermograms obtained for the NiFeGa specimen subjected to 1st cycle of compression is presented in Fig. 2; when a) shows temperature distribution from the specimen surface recorded during loading, b) after loading to the reversible strain limit 2%, c) during the unloading while the reverse transformation takes place. One can notice that the temperature distributions observed on the specimens surface during the loading and unloading processes is not uniform. The inclined band of higher temperature shown in Fig. 2a is related to the exothermic martensite transformation, while the band of lower temperature inclined in the opposite direction presented in Fig. 2c, is related to the endothermic reverse transformation. It can be concluded that the martensite forward and reverse transformation occurs in heterogeneous way by nucleation and development of the new phase.

3.2. Mechanical and temperature characteristics obtained for FSMA subjected to compression with a pause between subsequent cycles

The compression test was carried out in a way that after each cycle of loading-unloading there was a pause until the temperature regained its initial level. Thus, it can be assumed that before each cycle of deformation the FSMA specimen regained its initial thermodynamic state. Comparison of the stress-strain curves obtained for 6 subsequent cycles of compression polycrystalline NiFeGa within the strain range 2% is presented in Fig. 3a, while their related temperature changes obtained as average from the specimen surface are presented in Fig. 3b respectively.

One can notice looking at Fig. 3 that the maximal stress and the maximal temperature changes increase in the subsequent compression cycles. It was probably caused by the crystallographic defects, developing in the specimen during the FSMA cycling. In order to analyze the FSMA cycling behavior, comparison of the stress-strain curves obtained for 1st (a) and 6th (b) cycles of compression of the polycrystalline NiFeGa within the strain range 2% and pauses introduced between the subsequent cycles, are presented in Fig. 4.

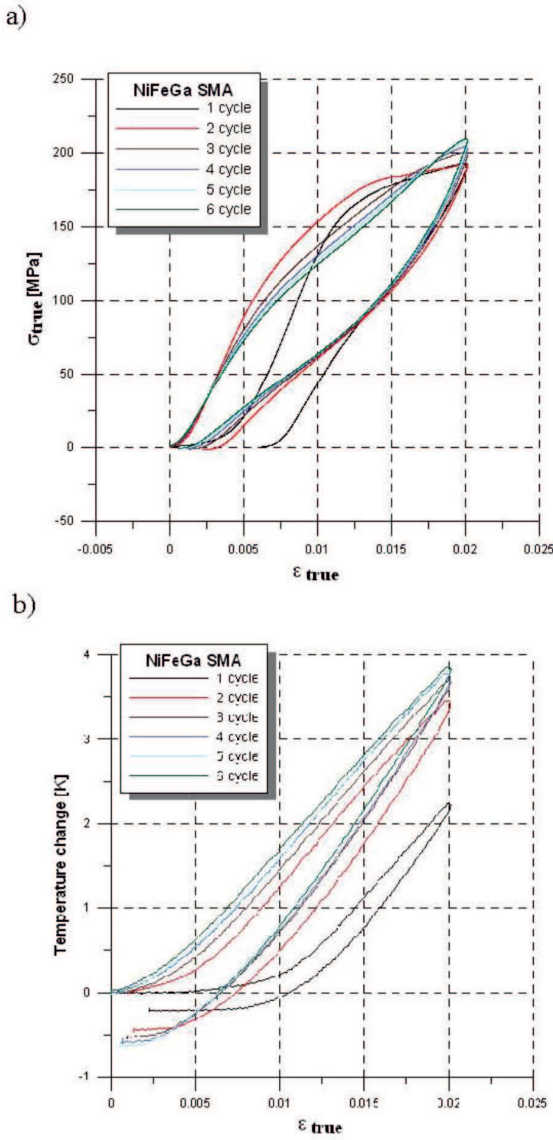


Fig. 3. Stress-strain and temperature-strain curves obtained during 6 cycles of compression of NiFeGa

One can conclude from Figures 3 and 4 that for the subsequent cycles the stress-strain curve is developing and gradually narrowing. Moreover, the maximal achieved stresses increase from value of 194 MPa to 212 MPa, recorded for the 1st and 6th cycles, respectively. In turn, the maximal specimen temperature increases from 2,24 K to 3,85 K, recorded for the 1st and 6th cycles of the FSMA compression loading.

3.3. Mechanical and temperature characteristics obtained for FSMA subjected to direct loading-unloading compression cycles

In the following test the compression was carried out in a way that after the 1st complete cycle of loading and unloading the subsequent cycles were realized directly.

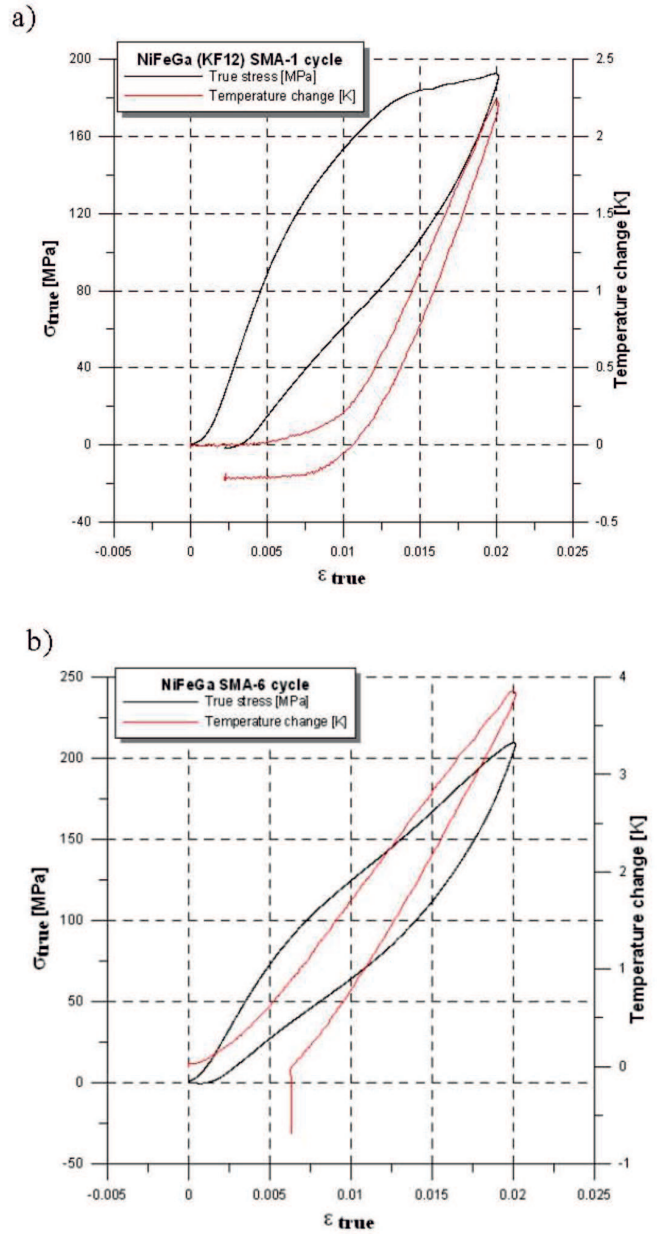


Fig. 4. Stress-strain and temperature-strain curves obtained for 1st and 6th cycles of compression polycrystalline NiFeGa with strain rate 10⁻²s⁻¹ and within the strain range 2%

Thus, a usual mode of the SMA actuator work without any break was under consideration. Comparison of the stress-strain curves obtained for 6 subsequent cycles of compression polycrystalline NiFeGa within the strain range 2% are presented in Fig. 5a, while their related temperature changes obtained as average from the specimen surface are presented in Fig. 5b, respectively.

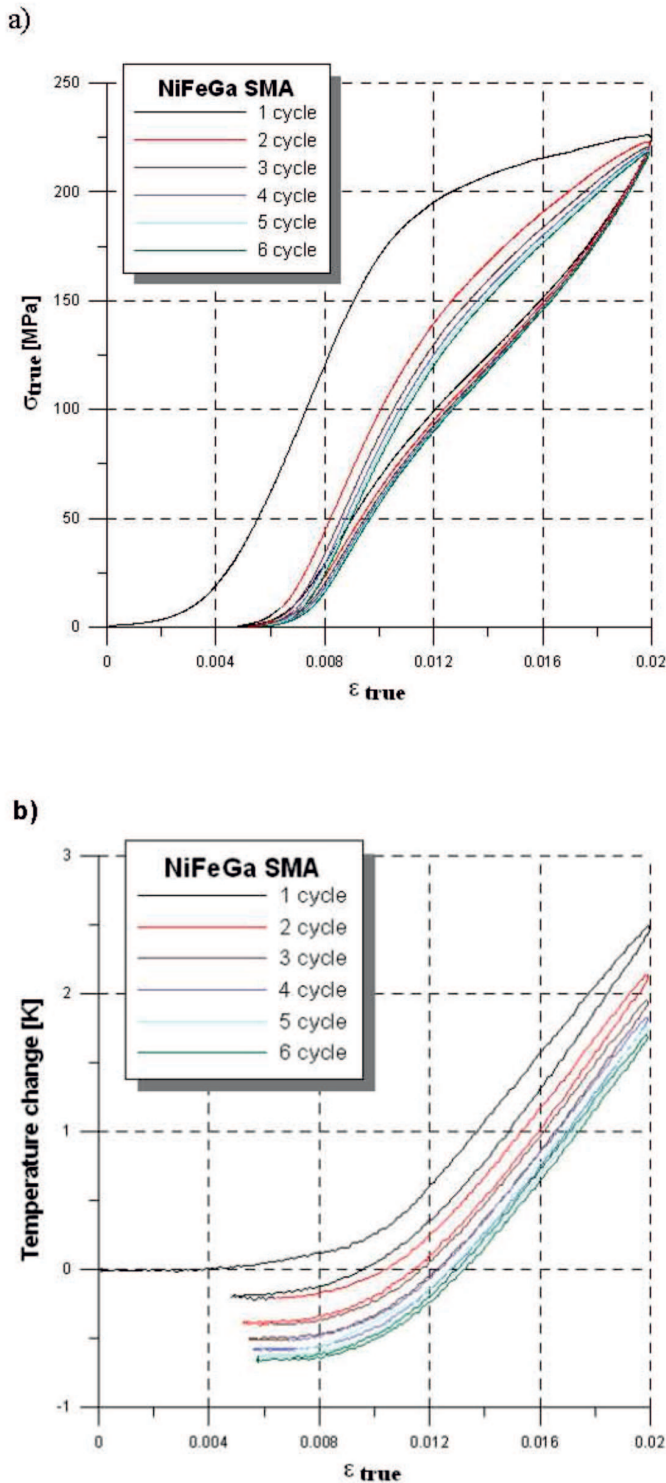


Fig. 5. Stress-strain (a) and temperature-strain (b) curves obtained for 6 direct loading-unloading compression cycles of NiFeGa

Looking at Fig. 5, one can notice that in the compression cycles realized directly the maximal stress level achieved and the maximal temperature changes decrease for the subsequent cycles of loading. It was caused by the fact that after each complete cycle of loading-unloading the specimen temperature drops below its initial temper-

ature level. Hence, the specimen temperature is lower at the start of the subsequent cycle of deformation. Such a specimen thermal behavior can be explained on the grounds of symmetry of the martensite forward and reverse transformations, confirmed by the SMA thermodynamic theory and the heat transfer rules. The lower strain rate, the higher temperature decrease after the pseudoelastic cycles of loading-unloading was recorded [9]. Comparison of the stress-strain curves obtained for 1st (a) and 6th (b) cycles of compression of the polycrystalline NiFeGa within the strain range 2%, realized directly, without a pauses between the subsequent cycles, are presented in Fig. 6, respectively.

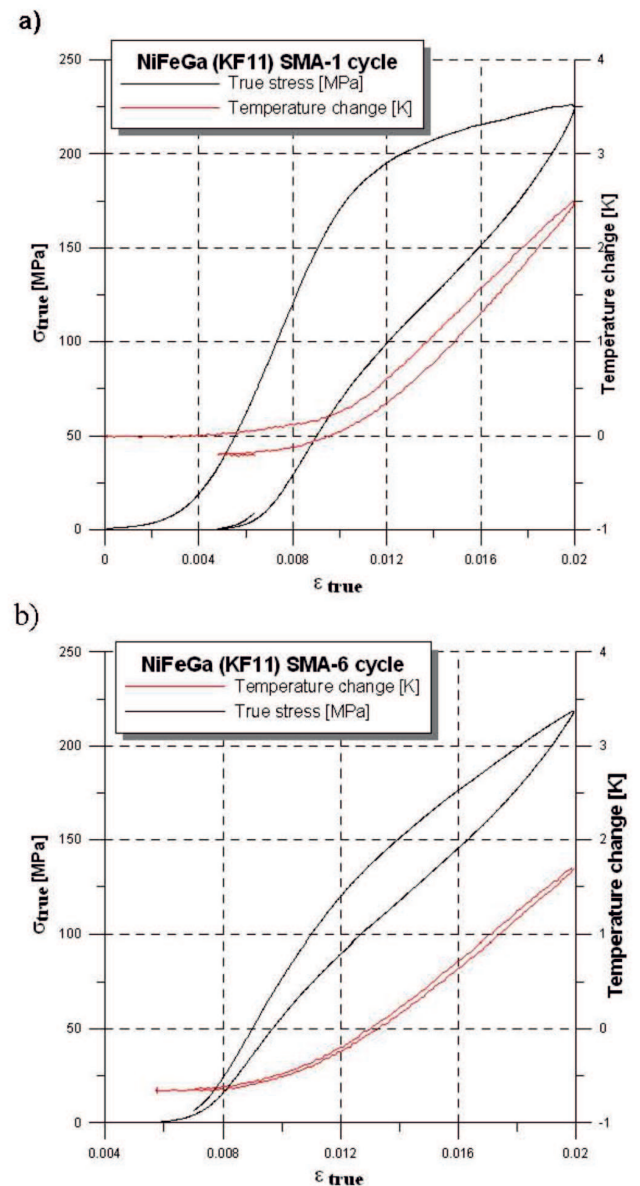


Fig. 6. Stress-strain and temperature-strain curves obtained for 1st and 6th direct cycles of compression polycrystalline NiFeGa with strain rate 10^{-2}s^{-1} and within the strain range 2%

One can conclude from the Figures 5 and 6 that for subsequent cycles realized directly the stress-strain curve is gradually significantly narrowing. Moreover, the maximal stress decreases from 226 MPa for the 1st till 218 MPa recorded for the 6th cycle of compression. In turn, the maximal specimen temperature related to them decreases from 2,5 K for the 1st till 1,7 K recorded for the 6th cycle of the FSMA compression loading.

4. Discussion

Comparison of the temperature changes vs. time, obtained for 6 subsequent cycles of compression polycrystalline NiFeGa performed with the strain rate of 10^{-2}s^{-1} and within the strain range 2%, realized in two approaches described above, are presented in Fig. 7.

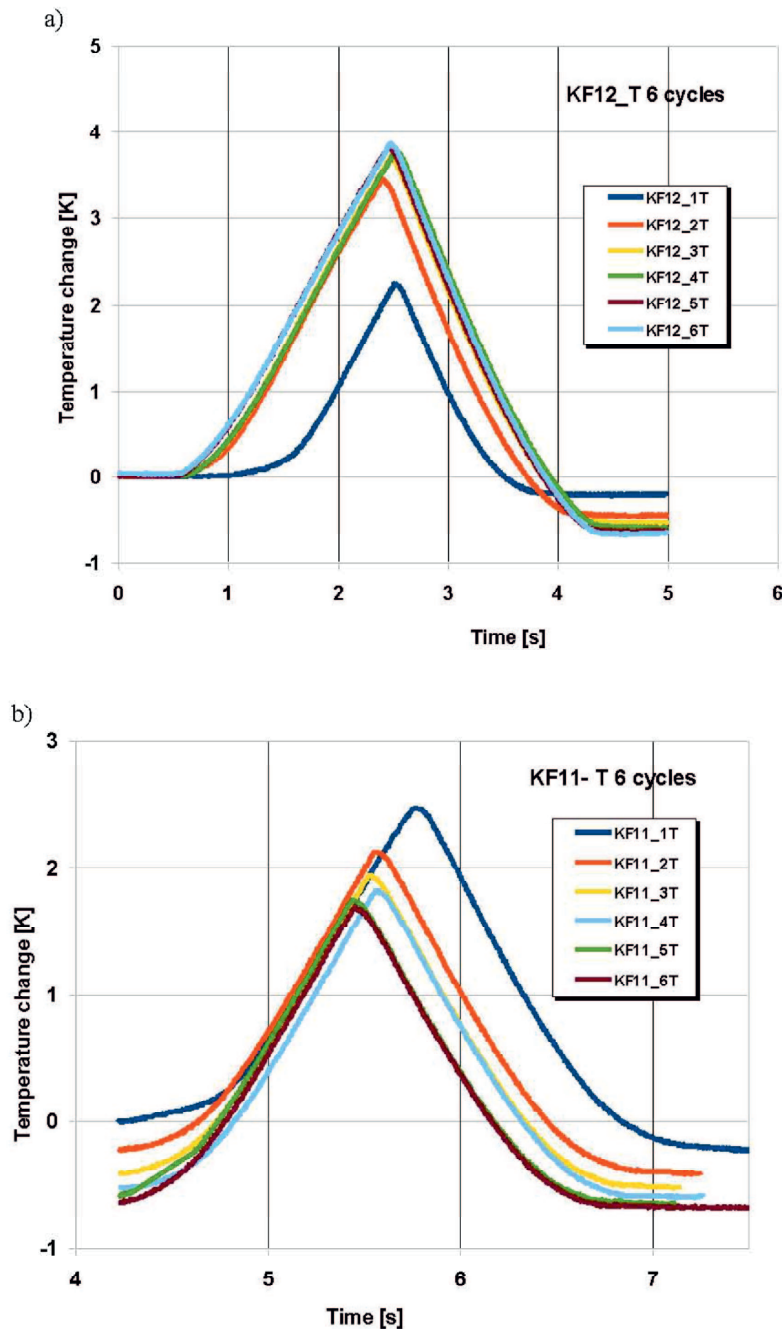


Fig. 7. Stress-strain curves of 6 subsequent cycles of compression polycrystalline NiFeGa within strain range 2%: a) with a pause between subsequent cycles; b) realized directly

Looking at the presented figures one can comment that the temperature changes accompanying the subsequent cycles of the forward and reverse transformation can characterize the FSMA thermomechanical behavior quite distinctly.

Comparing the stress-strain curves obtained during the polycrystalline compression realized with pauses (Figs 3, 4, 7a), it can be noticed that both the maximal stress and the maximal temperature changes increase for the subsequent cycles of loading. Since in this case the FSMA thermodynamic state is similar before start of each cycle, therefore the higher values of the recorded stresses and temperature changes were probably caused by the crystallographic defects induced and developed in the alloy during the specimen cycling loading. The number of defects – the obstacles on the transformation way - increases due to the cycling, so both the maximal stresses and the temperature values also increase for the subsequent compression cycles.

Comparing the stress-strain curves obtained during the polycrystalline reversible compression cycles realized directly, without pauses between the subsequent cycles (Figs 5, 6, 7b), one can notice that both the maximal stress and the maximal temperature change values decrease for the subsequent cycles of loading and unloading. Such behavior of the specimen maximal stresses and temperature achieved can be explained by the symmetry of the martensite forward and reverse transformations, confirmed by the SMA thermodynamic theory. One can conclude from comparison of Fig. 7 to Figs 3-6 that the temperature changes accompanying the forward and reverse transformation can characterize the FSMA much markedly and visible than the respect stress-strain curves.

5. Conclusions

As a result of the compression tests carried out for polycrystalline specimens of magnetically controlled shape memory alloy ($\text{Ni}_{54}\text{Fe}_{19}\text{Ga}_{27}$) it can be concluded:

1. The martensite forward and reverse transformation occur in heterogeneous way, since their related temperature distributions observed on the specimens surface during the loading and unloading processes were not uniform.
2. Values of the maximal temperature changes measured in contact less way by the fast and sensitive IR camera during compression of the specimens were in the range of 2,25 K- 4,5 K.
3. During the polycrystalline compression, realized with pauses, both the maximal stress and the maximal temperature increments increase for the subse-

quent cycles of loading. The higher values of the recorded stresses and the temperature changes were probably caused by the crystallographic defects, induced and developing in the alloy during the specimen cycling loading. For the compression cycles realized directly the maximal stress level achieved and the maximal temperature changes decrease for the subsequent cycles of loading and unloading, since the temperature after the unloading drops below the initial specimen temperature. This can be explained by the symmetry of the martensite forward and reverse transformations, confirmed by the SMA thermodynamic theory. The maximal discrepancies were found between the first and the second cycles of the NiFeGa compression. Like usually for shape memory alloys cycling, the higher number of the cycling, the lower discrepancies were observed. Therefore, in order to assure the FSMA sufficient reliability, an initial mechanical training of the SMA is recommended.

Acknowledgements

The financial support of the Polish Ministry of Science and Higher Education under Grant No 501220837 is greatly acknowledged. The authors are grateful to Prof. E. Cesari for supplying the FSMA specimens as well as for valuable advice, to Prof. H. Tobushi for scientific comments and to Dr. M. Maj for recording and elaborating the experimental infrared data.

REFERENCES

- [1] K. Ullakko, *Journal of Materials Engineering and Performance* **5**, 405-409 (1996).
- [2] K. Otsuka, C.M. Wayman (Eds.), *Shape Memory Materials*, Cambridge Univ. Press, Cambridge (1998).
- [3] E. Cesari, J. Pons, C. Segui, V.A. Chernenko, *Proceedings of the XIX Conference*, Cracow, Poland, ed by H. Morawiec & D. Stroz, 128-133 (2004).
- [4] Ma Yunqing, Yang Shuiyuan, Wang Cuiping, Liu Xingjun **58**, 918-921 (2008).
- [5] R. Santamarta, E. Cesari, J. Muntasell, J. Font, J. Pons, P. Ochin, *Intermetallics* **18**, 977-983 (2010).
- [6] J.A. Shaw, S. Kyriakides, *Acta Mater.* **45**, 2, 683-700 (1997).
- [7] E.A. Pieczyska, S.P. Gadaj, W.K. Nowacki, H. Tobushi, *Bull. Pol. A. Sci Tech Sci* **52** (3), 165-171 (2004).
- [8] S.P. Gadaj, W.K. Nowacki, E.A. Pieczyska, H. Tobushi, *Archives of Metallurgy and Materials* **50**, 661-674 (2005).
- [9] E.A. Pieczyska, S.P. Gadaj, W.K. Nowacki, H. Tobushi, *Experimental Mechanics* **46**, 4, 531-542 (2006).

- [10] S. Daly, G. Ravichandran, K. Bhattacharya, *Acta Materialia*, **55** (2007).
- [11] E.A. Pieczyska, H. Tobushi, S.P. Gadaj, W.K. Nowacki, Sakuragi Toshimi, *Materials Transactions* **48**, 10, 2679-2686 (2007).
- [12] E.A. Pieczyska, *Pomiary Automatyka Kontrola* **55**, 958-961 (2009).
- [13] E.A. Pieczyska, *Journal of Modern Optics* **57**, 18, 1700-1707 (2010).
- [14] S.P. Gadaj, W.K. Nowacki, E.A. Pieczyska, *Infrared Physics and Technology* **43**, 151-155 (2002).
- [15] D. Favier, Y. Liu, L. Orgeas, G. Rio, *Proc. JUTAM Symposium on Mechanics of Martensite Phase Transformation in Solids*, ed. By Q.P. Sun, 205-212 (2001).
- [16] E.A. Pieczyska, S.P. Gadaj, W.K. Nowacki, J. Luckner, H. Tobushi, *Strain* **45**, 93-100 (2009).
- [17] H. Tobushi, E.A. Pieczyska, W.K. Nowacki, Y. Sugimoto, *Solid State Phenomena* **154**, 47-52 (2009).
- [18] H. Tobushi, E.A. Pieczyska, W.K. Nowacki, T. Sakuragi, Y. Sugimoto, *Archives of Mechanics* **61**, 241-257 (2009).
- [19] H. Tobushi, E.A. Pieczyska, W.K. Nowacki, K. Date, K. Miyamoto, *J. Theoret. Appl. Mechanics* **48**, 1043-1056, 4 (2010).
- [20] E.A. Pieczyska, H. Tobushi, K. Date, K. Miyamoto, *JSME International Journal Series a-Solid Mechanics and Material Engineering* **4**, 1-9, 8 (2010).
- [21] E.A. Pieczyska, R.B. Pęcherski, S.P. Gadaj, W.K. Nowacki, Z. Nowak, M. Matyjewski, *Arch. Mech.* **58**, 3, 273-291 (2006).