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Phase Analysis of NiTi Shape Memory Wires and Computer Simulations of the Superelastic Effect

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Shape memory alloys belong to the group of intelligent materials due to their exceptional properties related to reversible martensitic transformation. They show both superelasticity and shape memory effects. The modelling of the superelastic phenomena occurring in NiTi alloys is important due to its application in the further development of materials for dedicated applications. A computer simulation of static tensile test was carried out using the finite element method for NiTi wire and obtained results were compared with experimental curve. For the determination of phase composition at ambient temperature and courses of phase transitions during cooling and heating the X-ray powder diffraction and differential scanning calorimetry methods were used. X-ray diffraction phase analysis exhibited that the studied wire at room temperature has $B2$ — parent phase structure. Additionally, differential scanning calorimetry measurements showed that the phase transformations during cooling and heating occur in a temperature range of about −20 to +30 °C.

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1. Introduction

Shape memory alloys (SMAs) belong to the group of intelligent materials due to their exceptional properties related to reversible martensitic transformation, such as superelasticity and shape memory effects. The phenomenon of superelasticity is the recovery of an original shape during a mechanical loading-unloading cycle at a constant temperature, while shape memory phenomena occur as a result of temperature and stress operation.

Shape memory alloys are promising materials for innovative applications in numerous areas of industry. NiTi shape memory alloys of near equiatomic chemical composition with technological name NITINOL are found in numerous applications in medicine. Unique properties of these alloys contribute to miniaturization of medical instruments, reduction of treatment invasiveness, simplifying operations and shortening hospitalisation times. These alloys feature a very good biocompatibility and biotolerance, which means that they are used amongst other materials for stents, braces, Amplatzer Septal occlusion devices, and staples for osteosynthesis [1–3].

NiTi alloys are extensively studied experimentally in terms of mechanical behavior and thermal properties and alternative is the numerical calculations to simulate the behavior of these alloys using the finite element method (FEM). Computer simulations allow to test different processes without the involvement of the resources. This allows to perform expensive and time-consuming measurements in a "virtual" way saving financial resources and the time required for preparing prototypes and conducting the actual tests [4].

The ANSYS software belongs to the group of most advanced FEM computational systems, enabling the user to carry out linear and non-linear analysis. The package also offers functions allowing interactions, e.g. structural-flow or thermal-mechanical analysis. This environment enables quick performance and unequivocal analysis of phenomena that occur in materials and complex microstructures, including SMAs.

In this paper we present the results of numerical computations using the finite elements method in the ANSYS software of selected NiTi wire exhibiting superelasticity. In this manner the superelastic behavior of NiTi wire was tested under the influence of the various external forces causing initial martensitic transformation in results of the static tensile test. This allows us to predict the behavior of the tested wires under the influence of a given value of external force acting along the cross-section of studied sample. Static tensile test allows to determine the stress levels at the beginning and at the end of austenitic and martensitic transformations. These parameters were necessary during numerical calculations of superelasticity phenomena in studied NiTi alloy using ANSYS software. The results of FEM analysis have been compared with the experimental results of static tensile test.

Moreover, this paper provides the results of experimental research, including X-ray diffraction analysis to obtain a phase composition of studied alloy at ambient temperature and differential scanning calorimetry (DSC) measurements to obtain characteristic temperatures of austenitic $\rightleftarrows$ martensitic transformation.

2. Material and methods

The material used in our studies was commercial NiTi alloy with nearly equiatomic chemical composition produced by Euroflex Company. The alloy exhibits an excellent superelastic effect. The specimen was 150 mm long wire of 1.2 mm diameter. Experimental methods such as DSC, X-ray powder diffraction (XRD), scanning electron

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microscopy (SEM), and static tensile tests were used to characterize the material.

DSC measurements were carried out on a DSC 1 Mettler Toledo in the temperature range from −120 °C to +120 °C with 10 °C/min heating and cooling rate. From every studied wire a piece was cut and DSC measurements were performed.

The X-ray diffraction studies were carried out using a Philips PW1130 diffractometer with Cu K_α radiation collection over a wide angular range (10−130°). XRD patterns were identified using the PDF4 database (NiTi $B2$ phase (CsCl type), ref. code 01-076-3614).

Measurements of SEM with electron back scattering diffraction (EBSD) were carried out on flat samples metallographically prepared from round wires. Measurements were performed using JSM-6480 microscope with JEOL EBSD detector.

The tensile tests were performed on an Instron 5982 L2127 testing machine with a Bluehill operating system. The test was conducted in 3 cycles and the specimen was expanded to 10% of relative length.

For the numerical analysis a simple geometry (wire) was chosen which allows straightforward generated mesh. Additionally, the choice of geometry was determined by the possibility of carrying out experimental tests, which were performed for the needs of this study.

3. Results and discussion

The first step of performed work was to characterize the material used for further studies. For numerical modelling it is essential to know the structure of the modelled material, because the phase composition is significant for the mechanical properties and characteristic parameters of studied materials. In order to determine the characteristic transition temperatures of the austenitic and martensitic phase transitions and to verify the suitability of the studied NiTi alloy for medical implants DSC measurements were performed. Figure 1 presents the results of DSC measurements for samples of NiTi wire with diameter of 1.2 mm during cooling and heating cycles.

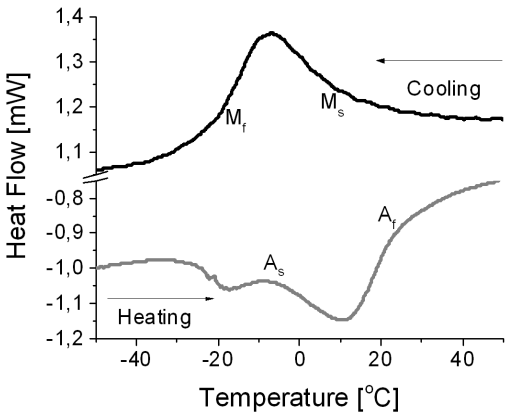

Fig. 1. DSC measurements for samples of NiTi wire of 1.2 mm diameter in the temperature range from −120 °C to +120 °C with 10 °C/min heating rate.

As seen in the figure the phase transformations during cooling and heating occur in a temperature range of approximately −20 to +30 °C. The obtained data confirm that the tested NiTi alloy can be used in the manufacture of medical implants [5]. DSC measurements showed the M_{sm} temperature below 10 °C, thus the studied material exhibits parent phase (austenite) at the room temperature.

In order to confirm the CsCl-type structure at the room temperature XRD measurements were performed. Figure 2 presents the X-ray diffraction pattern of NiTi sample with 1.2 mm diameter at the room temperature.

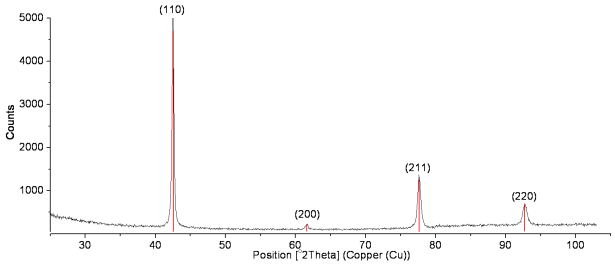

Fig. 2. X-ray diffraction pattern of the NiTi sample at room temperature.

Analysis of the diffraction pattern indicates that the studied alloy possesses the CsCl-type structure — only characteristic peaks for the austenite phase are observed. Therefore it was assumed for the numerical design that the NiTi wire is in the austenitic phase [6].

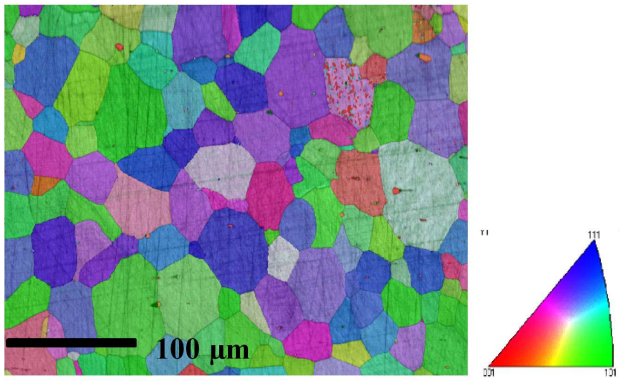

Fig. 3. EBSD map of NiTi sample of 1.2 mm diameter showing shape and orientation of the NiTi austenite grains at room temperature.

The presence of the austenite phase in the studied materials has been also confirmed by EBSD method and the results are shown in Fig. 3. In the figure there can be seen the shape of grains at room temperature in the studied NiTi wire. The color of grains depends on the orientation of the grains relative to some defined axis. The studied wires do not possess any texture — the observed crystallographic orientation of grain is uniformly distributed.

Figure 4 shows a comparison of experimental data of static tensile test with curve obtained by FEM calculations for NiTi wire of 1.2 mm diameter. Methodology of numerical calculations by the finite element method is given in work [7].

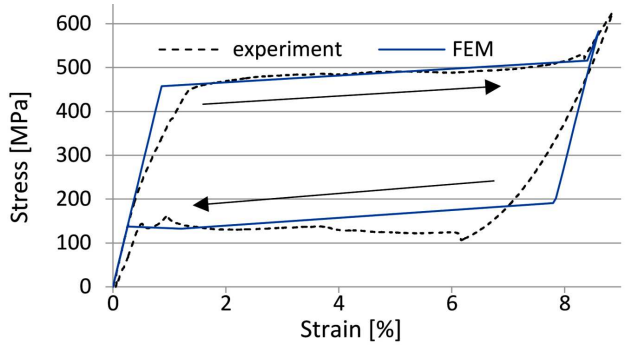

Fig. 4. Comparison of numerical simulation results (line) with experimental data (dots) of the tensile test for NiTi wire of 1.2 mm diameter.

As seen from the figure under uniaxial load, NiTi wire extends nearly by 9% reaching a maximum stress of 600 MPa. Similar results were obtained by Auricchio et al. [8]. However most of the time the upper plateau reaches level of stress about 500 MPa and at the end of austenitic transformation increases to 600 MPa.

On the basis of the comparison between experimental results and computer simulations it can be concluded that a good visible match was obtained. It provides a basis for further exploration in the field of numerical modeling of NiTi alloys.

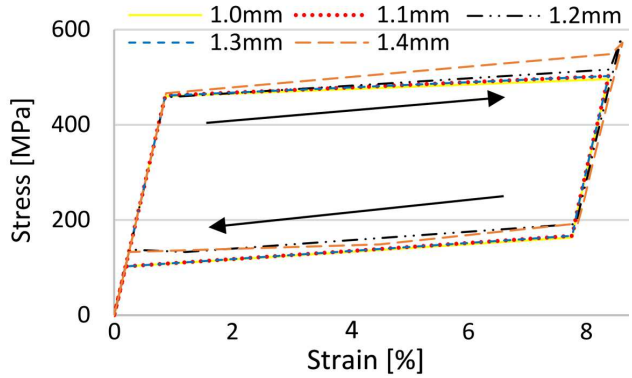

Fig. 5. Computer simulations of the superelastic effect of the tensile test for NiTi wire of 1.0–1.4 mm diameters.

Figure 5 presents the results of five numerical simulations for different diameters (1.0–1.4 mm) NiTi wires. During the numerical design, only the values of the force have been changed. The value of force increased together with increase in the diameter of the NiTi wire in order to achieve the same stress. As seen in Fig. 5, there was obtained a very good convergence of simulation results of static tensile test for diameters of 1.0–1.4 mm. This proves the correctness of parameters and numerical design.

4. Summary

X-ray diffraction analysis indicates that the studied alloy has $B2$ (CsCl-type) structure because only characteristic peaks of this phase were observed on the diffraction patterns recorded at a room temperature (Fig. 2).

DSC measurements showed the presence of austenite–martensite transformation at temperature below 10 °C confirming presence of parent phase at the room temperature (Fig. 1).

The presence of the austenite phase in the studied materials has been also confirmed by EBSD method and the results are shown in Fig. 3.

The assumption in numerical analysis that tested samples were in austenite phase were correct and confirmed by experimental measurements.

The superelastic behaviour of NiTi alloy was simulated based on a 3D simulation of static tensile test. Computer simulations were performed using finite element method for NiTi wires of 1.2 mm in diameters using ANSYS software.

A relatively good agreement between the computer simulation results and the experimental data has been obtained. Numerical simulations allow to estimate the forces acting on NiTi wire during tensile without the need for expensive and time-consuming experiments. A good agreement between obtained simulation results and experimental data justify the use of the FEM to study the behavior of a superelastic NiTi alloys. The comercial ANSYS package allows to obtain an effective simulation of superelastic behaviour of NiTi.

Analysis of superelastic behavior of NiTi alloys will be continued in order to investigate necessary parameters of these alloys, needed during the numerical design. For this purpose 3-point bending tests and computer simulations will be carried out. The elaboration of modeling methodologies of superelastic behavior of NiTi wires allows further numerical simulations of more complex elements. It would help to eliminate or at least minimize the number of necessary experimental tests.

References

[1] L.G. Machado, M.A. Savi, *Brazil. J. Med. Biol. Res.* **36**, 683 (2003).

[2] T.M. Mereau, T.C. Ford, *J. Am. Podiatr. Med. Assoc.* **96**, 2 (2006).

[3] C. Suárez, T. Vilar, P. Sevilla, J. Gil, *Int. J. Corros.* **2011**, 482485 (2011).

[4] D. Tarniţă, D.N. Tarniţă, D. Popa, D. Grecu, R. Tarniţă, D. Niculescu, F. Cismaru, *Roman. J. Morphol. Embryol.* **51**, 145 (2010).

[5] K. Nosalik, M. Kowala, *Dent. Med. Probl.* **49**, 433 (2012).

[6] X. Xu, M. Eng, Y. Zheng, D. Eng, *J. Endodont.* **32**, 4 (2006).

[7] B. Młocek, *Modell. Eng.* **21**, 433 (2014).

[8] F. Auricchio, R.L. Taylor, J. Lubliner, *Comput. Meth. Appl. Mech. Eng.* **146**, 281 (1997).