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## METHOD OF PROGRAMMING THE NITINOL SPRINGS IN THE SPACE OF THE KILN CHAMBER

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### METODA PROGRAMOWANIA SPRĘŻYN Z NITINOLU W PRZESTRZENI KOMORY PIECA

#### Abstract

This paper shows a method of programming the NiTiNol springs by heating them up in the furnace chamber filled with technical nitrogen. A fully specified methodology, which consists of preparing the spring forms, the numerical analysis of the heating time and the description of the heating process are presented in this article. The effectiveness of the following method is confirmed using research of shape retention after a series of duty cycles performed by an activated NiTiNol spring.

**Keywords:** NiTiNol, Shape Memory Alloys (SMA), NiTiNol programming, helical springs

#### Streszczenie

W artykule opisano sposób programowania sprężyn z NiTiNolu z wykorzystaniem pieca z atmosferą azotu technicznego. W ramach artykułu opisano metodykę procesu. W jej skład wchodzi metoda przygotowania form ze sprężynami, obliczenia numeryczne początkowych nastaw pieca oraz opis procedury wygrzewania w atmosferze azotu. Praca została podsumowana badaniami odwzorowania kształtu sprężyny po procesie programowania.

**Słowa kluczowe:** NiTiNol, materiały z pamięcią kształtu, programowanie NiTiNolu, sprężyny

## 1. Introduction

Shape Memory Alloys (SMA) are a group of materials which can be classified as smart materials [1-3]. These materials have the ability to recreate a programmed shape as a result of certain external factors e.g. temperature, magnetic field or a previously applied stress. One of the most commonly used shape memory alloys, along with alloys based on copper and iron, is NiTiInol. Originally it was invented by Bühler in 1962 [3] and patented along with Wiley, because of the characteristic mechanical memory, in 1965 [2]. NiTiInol is an alloy of nickel and titanium (NiTiX). Depending on the source, the amount of nickel in an alloy is in a range of 53-57% wt. [1-3], or 53-54% wt. [2].

NiTiInol has two stable phases: austenite with the cubic structure of the crystal (high-temperature phase) and martensite with the prismatic structure of the crystal (low-temperature phase). The martensite structure can be deformed without breaking its atomic bond – this phenomenon is called twinning [1].

The effect of the unidirectional shape memory in this material is based on the single time preservation of the programmed geometry. The essence of this process is to anneal a formed element, made of NiTiInol, which is currently in a high-temperature phase. The annealing is made in temperature 500°C within 30 minutes' time [1] and it is commonly called „programming” of the SMA. The parameters which can be used in the annealing process are also described in the following papers [6-8].

The heating time depends on the geometry of the programmed elements. It can be between 1 and 20 minutes. The exact value of the heating time can be determined thanks to the experimental studies based on the chemical composition of NiTiInol alloy [5-8].

Overheating of the NiTiInol alloy to temperatures higher than 600°C is not recommended due to the loss of chemical stability. It also has a negative effect on the oxidation resistance, because of damaging the thin layer of oxides which covers an alloy in normal conditions [4]. Upon heating the NiTiInol wire from temperature  $A_s$  (activation of the transformation) to  $A_f$  (end of a transformation), a transformation from martensite to austenite can be observed. Further increase of the temperature does not change the phase of an alloy, but causes the ordering of the crystal lattice and lowers the value of the internal stress. After the annealing process finishes, followed by a cooling process, the austenite phase stable in higher temperature with a crystal lattice of higher symmetry transforms into the martensite phase stable in lower temperatures, which is characterised with lower symmetry. In normal air temperature, a crystal lattice of the martensite phase is deformed due to twinning [1].

## 2. Methodology of the programming process

In order to obtain a desired shape of the NiTiInol alloy, it is necessary to increase temperature over the austenite transformation temperature during the programming process. The next step is to anneal an alloy in approximately 500°C in a specified time.

In this research program, two stages of forming and programming the shape details of helical springs were considered. Because of the straight shape of the NiTiNol wire provided by the distributor and used in this research, it was necessary to design a novel method of forming and securing the coiled springs. Otherwise they would lose their geometry during the heat treatment due to internal stress.

During the methodology studies, numerical analyses were made in order to determine the values of programming time and temperature. These calculations provided necessary data for selecting the proper heat treatment ramp and adjusting it to the performed programming process.

### 2.1. Shaping the helical spring

One of the most important stages of the programming methodology is shaping the NiTiNol helical spring. In order to complete this task, the dedicated apparatus was designed and manufactured. The designed device allows to shape a helical spring as well as to secure its geometry during heat treatment. Its construction and the methodology used were later described in the patent application [4]. The schematic of a device for manually shaping NiTiNol helical springs is presented in the Figure 1.

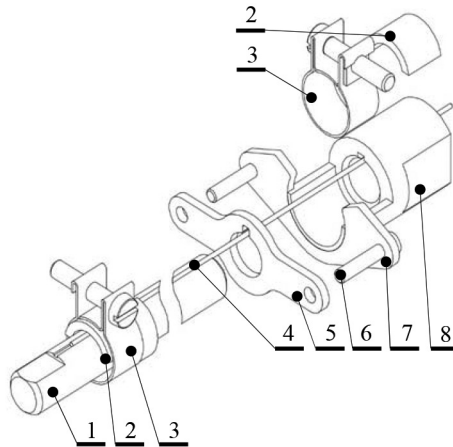


Fig. 1. Components of the device for manually shaping NiTiNol helical springs [4]: 1 – core, 2 – clamping pad, 3 – U-bracket, 4 – NiTiNol wire, 5 – spinning washer, 6 – blocking pins, 7 – pressure plate, 8 – base

The procedure of shaping NiTiNol helical springs for the programming process in a hot air stream using the previously described device consists of several elementary steps. Firstly, it is necessary to mount the bearing set in the vice using flat surfaces in the base of the device 8. Before the threading of the NiTiNol wire 4, U-bracket 3, which is open, must be embedded on the bearing set in such a way that its screw is placed in a relief in the base. Next, the wire is threaded through a bearing set and placed in the groove in the base. The spinning washer 5 is now mounted on the wire and then the remaining part of the wire is embedded in the

groove on the core 1. It is important to place it on the shorter shaft neck of the core, which is ended with the collar. To prevent movement of the wire, it is compressed using the clamping pad 2 and the U-bracket. After that, the spinning washer is fitted with pins 6, which block its rotational degree of freedom, and the wire lays in the grooves of axially mounted components. The core is translated through the centre holes in the spinning washer and the base until its collar has contact with the spinning washer, which is pressed against the pressure plate 7. The last stage is to put the clamping pads between the wire and the U-bracket, which is tightened up by a screw.

After the preparation stage, the coiling can start. First, the core is clutched with the proper tool by using flat surfaces on the core. Then the core is rotated along with pressing it with an axial load in the direction towards the bearing set. Each rotation creates one coil of a helical spring and causes the distance between the collar and the spinning washer to grow. When the spring is ready, it is necessary to clamp its end with the U-bracket, pull out the core from the bearing set and cut off the remaining wire.

The effect of the above-mentioned procedure is the NiTiInol helical spring, which is properly shaped, protected and ready for the programming process by putting it in a furnace chamber. An example of the prepared specimen is presented in Figure 2. The NiTiInol alloy used in the presented research had 55/45%wt Ni/Ti.

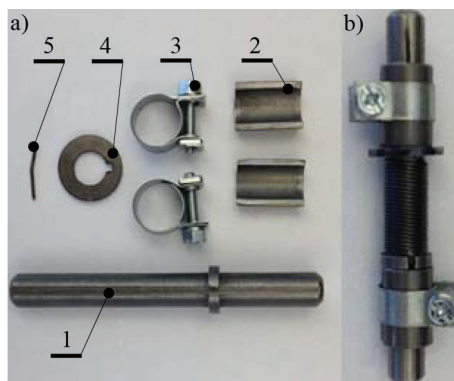


Fig. 2. Specimen ready for programming: a) components; b) form with coiled spring; 1 – core, 2 – clamping pad, 3 – U-bracket, 4 – spinning washer, 5 – NiTiInol wire

## 2.2. Simulation analysis of the heating process

A convenient way to determine the heating time in which a specimen will achieve the specified temperature is to perform the simulation in Abaqus – educational version. The obtained results are not precise due to the limit of nodes, but are adequate to design the programming process. An example of the results is presented in Figures 3 and 4 (red – 500°C, yellow – 350°C).

The characteristics of temperature of the NiTiInol spring in the function of the heating time is presented in Figure 5. Based on the performed simulations it can be concluded that the heating time for programming the NiTiInol spring at 500°C equals approximately 16 minutes.

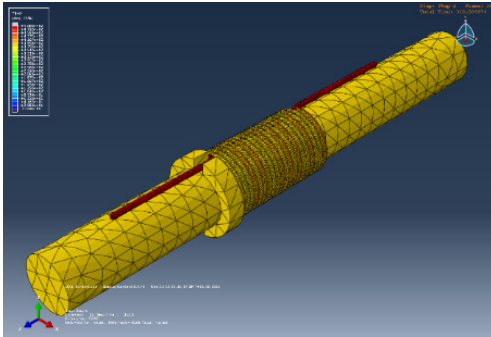


Fig. 3. Temperature distribution after 5 minutes of heating up in the furnace chamber at 500°C

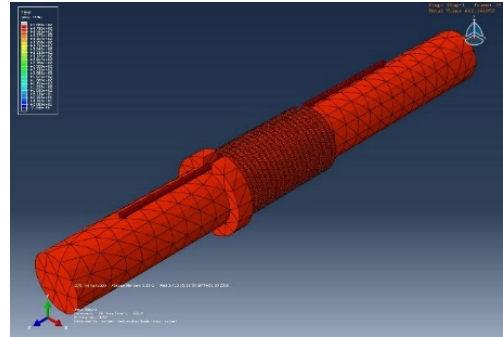


Fig. 4. Temperature distribution after 10 minutes of heating up in the furnace chamber at 500°C

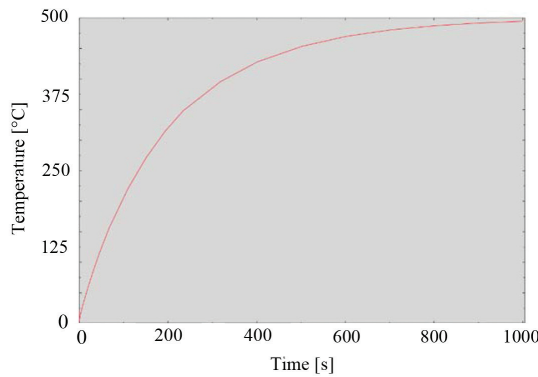


Fig. 5. Temperature characteristic of the NiTiNol spring in the function of the heating time

## 2.2. Industrial furnace characteristics

The NiTiNol alloy was heat-treated using the industrial furnace of VTR type shown in Figure 6. Multiple operations can be made in this furnace chamber e.g. tempering, annealing, heating and nitriding.

The work chamber of the furnace is made of heat resistant steel and has a cylindrical shape with vertical loading of the furnace feed. Circulation of gases inside the work chamber is forced by a fan, a baffle and a guide wheel. That is why a gas flows through the furnace similarly to the flow in the reversing chamber [1].

The industrial furnace is equipped with a resistance heating device. It's maximal operating temperature equals 650°C. It allows to realise thermal processes with a pressure ranging from 28 to 32 mbar. The allowable mass of components in the furnace cannot be higher than 200 kg and their dimensions cannot exceed 400 mm x 400 mm x 600 mm. The following gases can be used in the furnace chamber: nitrogen (neutral gas used for rinsing a chamber before and after nitriding), ammonia (used for nitriding) and air (activator of surfaces of treated elements). The measuring system makes it possible to control temperature of the furnace feed by a set of thermoelements (9 thrown-in thermocouples and 1 retort thermocouple). It is also possible

to analyse the chemical composition of the chamber atmosphere or check the pressure inside the furnace chamber during the heating treatment. The heating system makes it possible to define the heating ramp, which is used during annealing NiTiInol springs.



Fig. 6. VTR type industrial furnace, Institute of Machines and Motor Vehicles, Poznan University of Technology

### 3. Characteristics of the programming process

In order to program NiTiInol helical springs used in this research, a proper procedure of for the heating process was required. The performed process indicates the necessity to specify the heating velocity, temperature of the process and the annealing time for each type of specimens. By using the neutral protective atmosphere of nitrogen, it was possible to safely slide in and out a single specimen into the furnace work chamber in the exact determined heating time. For all samples, the heating velocity of the kiln was constant and equalled  $8^{\circ}\text{C}$  per minute and the procedure was similar. Only two variables were present in the process: annealing temperature and time. The comparison between each process performed during this research is presented in Table 1. The lowest temperature of programming was established at  $400^{\circ}\text{C}$ , because only in higher temperatures does NiTiInol lose its initial straight shape and gains a new programmed shape of the helical spring coiled on the core.

Programming of NiTiInol springs was divided into several stages. First, the work chamber of the furnace was emptied in order to get rid of the air inside. After the vacuum inside the chamber was generated, it was filled with nitrogen and the furnace was heated up to a desired temperature of  $400^{\circ}\text{C}$ ,  $500^{\circ}\text{C}$  or  $600^{\circ}\text{C}$ . Total time needed to anneal a specimen is the sum of the following times: the period in which the kiln chamber attains the desired temperature, the time needed to heat up a specimen to the same value and the time of annealing a specimen in the desired temperature. The second value (16 minutes) was calculated based on the simulations in Abaqus, while the third time was chosen for analysis (2.5, 5 or 7.5 minutes). After finishing the annealing process, the final stage of cooling down to  $70^{\circ}\text{C}$  started. In order to achieve that, nitrogen was removed from the furnace chamber until reaching the pressure of 0.5-1 mbar. The arrangement of specimens inside the furnace is presented in the Figure 7.



Table 1. Parameters for programming (heat treatment) in an industrial VTR furnace

400°C		Temperature of the process		
		500°C	600°C	
Time needed to achieve desired temp. in a kiln chamber		2 h 01 min	1 h 55 min	1 h 53 min
Time needed to heat a specimen to a desired temp. [min]*		16		
Annealing time [min]	Specimen no. 1 on a rod	5		
	Specimen no. 2 on a rod	5		
	Specimen on a feed basket**	33	31	30
* – time counted from the moment of achieving the desired temperature in the kiln chamber				
** – time counted from the moment of achieving temp. lower by 5°C than the desired temp.				

Each process contained three NiTiInol springs with two of them mounted on the rods in the sample uptake port (Figs. 7a, c) and one lying on the furnace feed basket (Fig. 7b). The sample uptake port was used in the programming process as a two-way canal lock. That is why it was possible to move specimens in and out of the furnace chamber before and after finishing the heating treatment.

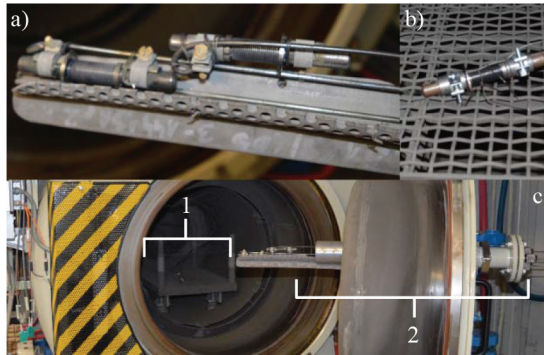


Fig. 7. Specimen distribution in the furnace during programming: a) specimens placed on a half cylindrical bonnet for a small feed; b) specimen placed on a feed basket; c) overall distribution; 1 – feed basket, 2 – canal lock in furnace

The task for the profile formed open bonnet (Fig. 7a) was to keep a proper path of motion for the specimen during putting it inside the kiln chamber. This bonnet prevented the sample from slipping outside the feed area during sliding it towards the outer rim of the kiln door. Thanks to that, it was possible to precisely slide the previously prepared NiTiInol springs on the forms inside the canal lock in the furnace door. The samples were mounted on bended

rods using extra burnt non-galvanised steel wire, which provided the specimen with position stability during the high-temperature heat treatment.

The described methodology makes it possible to put specimens inside the VTR furnace chamber, heat it to a desired temperature, anneal a sample for a specified amount of time and then pull out two of programmed springs outside the furnace chamber, all during a continuous heat treatment.

In order to control the heating velocity of the Ni-Ti wire, a thermoelement was mounted on its surface as shown in Figure 8. The retort thermocouple measurement was responsible for the steering process of the furnace chamber temperature, as per its calculation signal. Its value depends on the heating ramp, defined before the start as 8°C/min. Only the temperature directly on a coil of the spring from the feed basket was monitored. These time-based charts are presented in Figure 9.

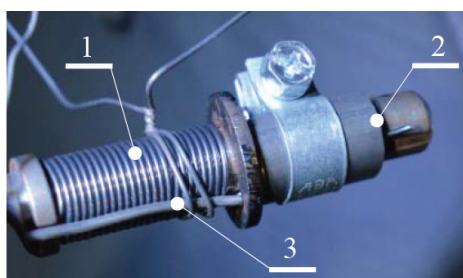


Fig. 8. Sample from the feed basket with a mounted thermocouple for temperature measurement: 1 – NiTiNol spring, 2 – programming form, 3 – thermocouple

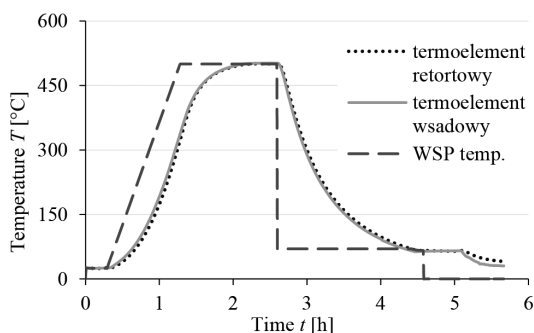


Fig. 9. Chart showing the progress of the NiTiNol helical spring programming. Process parameters: 500°C/5min – specimens on rods in the uptake sample port and 500°C/31 min – the specimen rested on the feed basket

The specimen, which laid on the feed basket, was heated from 26°C to 495°C for 2 hours and 1 minute. The last 5°C before reaching 500°C was gained in additional 10 minutes' period. Then, the annealing process was performed for 16 + 5 minutes – according to the research plan. In the final stage, the sample was cooled down along with the whole furnace chamber to 70°C. It lasted 1 hour and 49 minutes.



In the starting stage of cooling down, the coating of the furnace work chamber was refrigerated with forced air circulation. Then, in the later phase, when the door was opened, the temperature was reduced by convection. It is clearly visible in the chart in Figure 6 for the period of 5 hours and 6 minutes. After removing the specimens from the furnace, they were continuously chilled by convection with air.

#### 4. Method verification

The main goal of this research was to determine the influence of heat treatment parameters on the precision of the geometry of NiTiInol helical springs. The quality of the programmed springs was checked right after the programming process. The specimens were tested by loading them 36 times and measuring their length after activation with the electric current. In order to evaluate the presented methodology of programming, the results were compared with the spring programmed in a non-professional way. The reference specimen was programmed with the propane-butane gas torch by heating up the core with the spring in such a way that the inner cone was moved along the outer surfaces of the spring with a uniform distribution of the heat source. The whole process lasted 5 minutes and the temperature of the Ni-Ti wire was established at about 480°C.

For the comparison of the reference sample with the spring programmed in the furnace chamber, similar parameters were chosen: the programming temperature 500°C and the annealing time 5 minutes. This spring was mounted on the feed rod in the sample uptake port. As soon as the temperature in the chamber reached a desired value, the sample was slid into the chamber, where it was heated up for 16 minutes and then annealed for 5 more minutes. Then, the spring on the core was hidden back into the sample uptake port, where the cooling process took place at a temperature of approximately 100°C. Both samples are presented in Figure 10 (the reference sample) and Figure 11 (the tested sample).



Fig. 10. The spring programmed with the propane-butane gas torch: 5 min annealing time, ~480°C temperature, 21 m length of the spring after programming



Fig. 11. The spring programmed in the furnace chamber in the atmosphere of technical nitrogen: 5 mins annealing time, 500°C temperature, 22 mm length of the spring after programming

Taking into account the geometry of the NiTiNol springs removed from the cores, there is no difference between the reference and the test sample. Both ways of heating: in the furnace chamber and with the gas torch, reprogrammed the straight NiTiNol wire into the helical spring. This means that, in both cases, recrystallisation was performed successfully. An identical shape of the springs must be the effect of properly prepared forms.

A more important question was: how will the geometry of both springs change after a few duty cycles? In order to check that, it was necessary to deform the spring and then activate it, so that the spring should go back to the previous state due to the unidirectional shape memory effect. The test stand used in this research was designed and manufactured by the authors and shown in Figure 12. To achieve the strain of the spring, one end was fixed while the other end was loaded with a specified force. The loading force was obtained using a set of the twine and the hook weight. After the spring was extended to a proper value, it was activated by resistive heating with the electric current flowing through the coils of the spring. The electric power delivered to the sample equals about 22 W, which increases its temperature to 60-70°C. This temperature increase caused the austenite transformation in the wire, which led to the regularisation of the crystal lattice and the spring was compressed to its nominal length. During the experiment, the extension caused by the temperature and the force was measured. The view from the thermovision camera shown in Figure 13 illustrates the shortening of the spring due to the temperature increase.



Fig. 12. Test stand used to check the shape retention after duty cycles: a) before activation; b) after activation

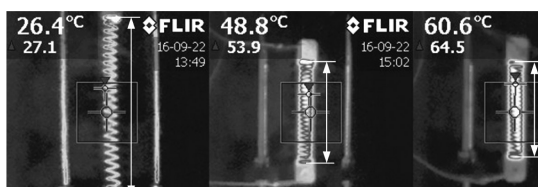


Fig. 13. Thermovision camera pictures during activation of the specimen loaded with 300 g and activated with 22 W power

Based on the series of performed experiments, elastic characteristics were drawn for two different methods of programming. They are presented in Fig. 14. The error for the displacement value was defined based on the standard deviation of the mean value of the estimator. When comparing the elasticity of both samples, it is clear that by using a furnace with nitrogen atmosphere inside the chamber to program the NiTiNol wire into the helical spring, the product

will have higher elastic constant. Both characteristics are linear, like in conventional helical springs, and this is confirmed by the value of  $R^2$  factor close to 1.

During the tests of the gas torch programmed spring, it was noticeable that its structure was destroyed due to the uncontrolled overheating while programming. It caused constant permanent deformations observed in the upper length of the spring after activation with the electric current. This means that the sample did not go back to its nominal length after taking the load off and final activation. The final form of the reference spring after 36 cycles of duty is presented in Figure 15a.

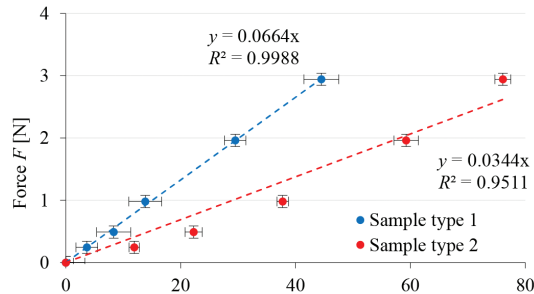


Fig. 14. Characteristics of the force in the function of displacement for the NiTiInol spring after activation for different loads

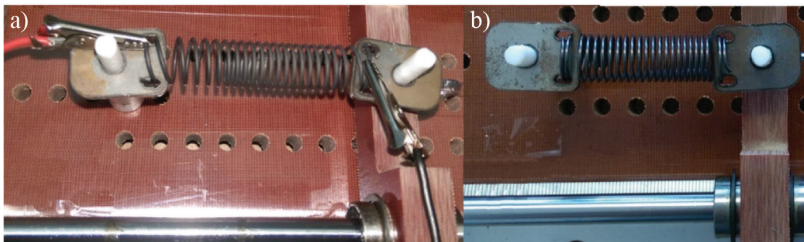


Fig. 15. Specimens after 36 cycles of loading and activation: a) spring programmed with the propane-butane gas torch, length before activation: 22 mm, length after 36 duty cycles and activation: 57 mm; b) spring programmed in the furnace chamber at 500°C for 5 mins, length before activation: 22 mm, length after 36 duty cycles and activation: 31 mm

The test sample failure occurred probably due to the simplified programming methodology. Programming with the gas torch is not a proper method. It results in an inhomogeneous distribution of heat in the various coils of the spring. Because of that, the crystal lattice of the alloy is unevenly reorganised. This can have a significant impact on the quality of the repeatability of the programmed shape. During the experiment, it was also noted that the electrical resistance of the test sample changes. The process progressed with the number of deformation cycles, which also can be explained with the inner structure failure.

The second test sample, which was annealed in the furnace chamber with protective nitrogen atmosphere had much better properties for shape preservation. The designed methodology provided the possibility for precisely steering the temperature and the programming time. Thus,

all the coils in the spring were properly annealed in the desired temperature thanks to preheating the specimen before the proper annealing. As the experiment shows, after 36 cycles of loading and activation, this specimen still maintains its initial shape after taking the load off (Fig. 15b).

## 5. Conclusions

The Industrial VTR furnace with the horizontal steel retort makes it possible to program the SMA Ni-Ti alloy in the protective atmosphere of technical nitrogen. By using the sample uptake port adapted to transport the springs coiled on the core with a specified geometry, it is possible to test a different annealing time in a single duty cycle of the furnace. The additional advantage of the designed methodology is the repeatability of the conditions in which the spring is programmed. It causes high stability and repeatability of the results obtained in post programming experiments. By using an industrial computer an ability to monitor and control the parameters of the performed heat treatment can be gained.

The reference sample programmed with the propane-butane gas torch revealed that this method is much less effective than the proposed methodology. Without the control of the programming parameters, it is impossible to obtain a unilateral temperature distribution in cross-sections of all coils of the NiTiInol spring.

During the experiments on the springs programmed in the furnace chamber, the unidirectional shape memory effect was observable. This phenomenon can be easily inverted and, as the test shows, it remains stable even for a series of duty cycles. This can be the determinant for the validity of the designed programming methodology with the VTR furnace.

In future research, focus should be directed onto a further analysis of the influence of time and form of the heat treatment on the properties of the final product – the helical NiTiInol spring.

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