A method of processing semi-finished products from an alloy of titanium nickelide TN-1

S V Kryuchkov¹, ⁴, V G Malinin², N P Bogdanov¹, M N Konovalov¹ and O A Malukhina³

¹ Ukhta State Technical University, 13, Pervomayskaya ul., Ukhta, 169300, Republic of Komi, Russia
² Orel State Agrarian University, 69, General Rodin ul., Orel, 302019, Russia
³ Yaroslav-the-Wise Novgorod State University, 41, B. St. Peterburgskaya ul., Veliky Novgorod, 173003, Russia

⁴ E-mail: kruk06@yandex.ru

Abstract. An experimental method was obtained for treating semi-finished products from titanium nickelide alloys TN-1 by twisting a cylindrical sample in the austenitic state at a temperature of $T = 493$ K to a predetermined angular strain, after which the sample was rigidly clamped, fixing the angular deformation of the sample, and cooled to a temperature of $T = 293$ K. At a given temperature, the rigid jamming of the sample is eliminated, while the isothermal return of the part of the deformation obtained by twisting in the austenitic state occurs. Then the sample is heated to a temperature of $T = 493$ K, and there is a shape memory effect (SME), manifested in the form of restoration of angular deformation. A systematic experimental study was carried out of the dependence of the influence of the complex regime of thermal-mechanical action containing the stages of generation and relaxation of stresses on the shape memory effect. It has been experimentally established that SME during heating after cooling in the direct MT range under pinched conditions can be observed with zero deformation effect in the final heating interval.

1. Research Methods
Non-traditional functional and mechanical properties of shape memory alloys (SMAs) are used to solve numerous practical problems: in power equipment, in robotics elements, in medicine, etc. [1]. The variety of various options for using materials with shape memory in industry makes it possible to talk about the relevance of the problem. In the vast majority of cases, materials when implementing reversible shape memory exhibit qualitatively similar behavior. The latter circumstance allows us to study the general laws of the effects of martensitic inelasticity on specific model materials, such as TiNi. Products from this material are difficult to manufacture. Therefore, the process of setting the desired shape for the semi-finished product is an important technological stage in the production of devices from alloys with the shape memory effect.

This article discusses a method for producing an SME by heating after cooling in the range of direct martensitic transformation (MT) in pinched conditions with zero deformation effect on the final heating interval.
Several methods are known for processing semi-finished products for materials with thermo-elastic martensitic transformations (TMT), which allow the formation of the shape memory effect (SME).

The first method is plastic deformation of the material by thermal cycling through the intervals of martensitic transitions of the TN-1 alloy under a constant voltage of various sizes [2].

The second method consists in plastic deformation of the material by thermal cycling through the intervals of martensitic transitions of the TN-1 alloy under constant voltage at the stage of cooling the material, and in the unloaded state at the stage of heating [3].

The closest analogue to the processing of semi-finished products, which is considered in this post, is the third method of processing semi-finished products from alloys with thermoelastic martensitic transformations – includes preliminary thermocyclic training of an alloy with thermoelastic martensitic transformations in torsion mode.

Thermal cycle mode: heating in the unloaded state in the temperature range from $T = 298K$ to $T = 500K$, and cooling to $T = 298K$ is performed under load [4].

Analyzing the data of three methods of processing materials with TMT, we can conclude that in all methods after training, there is a noticeable reversible deformation, manifested in the form of SME at the heating stage and its accumulation at the cooling stage, in the third method there were deformation effects associated with SME at heating in the unloaded state, with plasticity of direct transformation (PDT) during cooling under load through the interval of direct martensitic transformation (MT).

2. Research Results

The technical result of the experiment is to obtain an SME upon heating after cooling in the interval of direct MT in pinched conditions with zero deformation effect on the final heating interval.

This result is achieved by the method of processing semi-finished products from a titanium nickelide alloy TN-1 by pre-cooling and heating through the intervals of direct and reverse martensitic transformations in the torsion mode. The difference from the considered methods of processing materials is that in the austenitic state at a temperature of $T = 493 K$, the sample is isothermally twisted to a predetermined angular deformation value, after which the sample is rigidly clamped, fixing the angular deformation of the sample, allowing it to move freely in the axial direction. Then the sample is cooled to a temperature of $T = 293 K$, where the rigid jamming of the sample is eliminated. In this case, the isothermal return of the part of the deformation obtained by twisting in the austenitic state occurs. Subsequent heating of the sample to a temperature of $T = 493 K$ is accompanied by a shape memory effect, manifested in the form of restoration of angular deformation.

For testing, material was selected in the form of continuous cylindrical samples from an alloy close to equiatomic TH-1 with a length and diameter of the working part of 33 and 4 mm, respectively. The characteristics of temperature martensitic transitions were determined under torsion conditions at a shear stress of $\tau = 50 \text{ MPa}$ and amounted to $M_H = 350 K$, $M_K = 300 K$, $A_H = 360 K$, $A_K = 420 K$.

Before the test, the samples were annealed (in a muffle furnace in a special container) at a temperature of 723 K for one hour, followed by cooling to room temperature in air. The experimental part of the work was carried out on a special installation designed and manufactured at Ukhta State Technical University [5–7].

Before each experiment, 3–4 thermal cycles in the free state were carried out with each sample. The readiness of the sample for testing was evidenced by the absence of residual effects of reversible deformation on the resulting diagram $\gamma = \gamma (T)$.

The sample with the right end is fixed with two screws in the grip, rigidly connected to the right support. The left end of the specimen is fixed in the same way at the end of the shaft, which is able to freely rotate and move axially in the bearings of the bearings. The torque is transmitted through a pulley, rigidly fixed to the shaft by a keyed connection, on which a strong thread is wound with a load suspended at the end, while it is possible to change the direction of twisting of the load. The twist angle of the sample is determined by the scale of the measuring device. A computer program controls the measurement of the angle of rotation of the cross section of the sample $\phi$ and determines the relative twist angle according to the formula $\theta = \phi/l$. In this case, the accuracy of measuring the angle $\phi$ was
0.009 rad, and the torque was 0.01 N · m. The installation allows the sample to report a torque of up to 5 N · m. Heating was carried out by an electric furnace. The heating and cooling rate of the sample is set by the computer and is regulated by the sensor control program based on a calibrated chromel-kopel thermocouple, the junction of which was connected to the working part of the sample. To control the uniformity of heating the sample, two thermocouples were installed at different points of its working part. The sensor at a given heating rate records the temperature with an accuracy of 2 K.

The calculation results were considered as the average of the measurements for three samples made from the same bar with a relative error not exceeding 0.1%. When assessing errors, we took into account both random errors of multiple (obeying the Gaussian distribution) and errors of single (obeying the uniform distribution) measurements. To determine the total error of the result, the law of addition of independent quantities (errors) was used, which is also true for the addition of confidence intervals. Therefore, the confidence interval of the quantity $x$ measured in a series of experiments is written as follows,

$$
\Delta x = \sqrt{\Delta x_{re}^2 + \Delta x_{em}^2},
$$

where $\Delta x_{re}$ is the confidence interval corresponding to the random error of multiple measurements, $\Delta x_{em}$ is the confidence interval corresponding to the error of single measurements. For a confidence probability of $\alpha = 0.95$ and three times the measurement, the Student coefficient was chosen equal to a value of 4.3.

In addition to the integral characteristics of $M$ and $\varphi$, we used calculated estimates of the values of shear strains and shear stresses determined in the approximation of an ideal plastic body [8, 9] and assigned to the external fiber of the working part of the sample.

At the same time, the following working hypotheses were used as the basis for the estimated estimate:

1. The hypothesis of flat sections is fulfilled: the cross section of the beam – flat and perpendicular to the longitudinal axis, remains flat and perpendicular to the longitudinal axis even after deformation.
2. The radius drawn from the center of the cross-section of the beam after deformation remains a straight line (does not bend).
3. The distance between the cross sections, the diameters of the cross sections after deformation do not change, and the axis of the beam is not curved.

To obtain the result of an experimental study, a sample in the austenitic state at a temperature of 493 K was loaded with a given torque, setting the corresponding values of the tangential stresses and angular deformation.

The shear strain in the outer fiber was estimated by the formula (1):

$$
\gamma = (d \cdot \theta/2) \cdot 100 \% , \quad (1)
$$

where $\gamma$ is the angular deformation, $d$ is the diameter of the working part of the sample, $\theta$ is the twist angle. Moreover, the error in measuring shear strain did not exceed 0.05%.

According to [9], the estimation of the tangential stresses in the approximation of an ideally plastic body during torsion of continuous cylindrical samples was determined by the formula (2)

$$
\tau = 12 \cdot \frac{M}{\pi \cdot d^3}, \quad (2)
$$

where $M$ is the torque, $d$ is the diameter of the working part of the sample; $\tau$ – tangential stresses.

Next, the sample was rigidly clamped, with a fixed angular deformation of the sample using a device that works on the principle of a vise and consists of two flat jaws with an adjustment knob that moves the clamping screw. With it, the sponges diverge and contract, fixing the side surfaces of the pulley as tightly as possible (creating the effect of hard pinching of the sample). Elastic pads on the jaws evenly distribute the pressure force along the side surface of the pulley. The base of the device is attached using the adjusting screws to the bed of the installation, which allows you to configure the device for samples of various lengths. After this, the sample was cooled to a temperature of $T = 293$ K. At this temperature, the rigid jamming of the sample is eliminated, and a part of the deformation obtained by twisting in the austenitic state is isothermally returned. Then the sample in a free state is heated to a temperature of $T = 493$ K, and at the same time there is a shape memory effect, manifested in the form of restoration of angular deformation. In the coordinates of the angular deformation - temperature, the dependences of
the deformation responses for this method of processing the semi-finished product are presented (Figure 1). Here: $\gamma_A$ is the angular deformation due to the load of the sample at $T = 493$ K. $\gamma_m$ is the deformation due to unloading at $T = 293$ K. $\gamma_{SME}$ is the deformation due to the shape memory effect when heated in the temperature range 293–493 K.

**Figure 1.** Temperature dependence of angular deformation for loading stages: (1) loading in austenite to $\tau = 100$ MPa (a) and $\tau = 300$ MPa (b) and pinching; (2) – cooling in the temperature range 493 ÷ 293 K in a pinched state; (3) – removal of pinching condition; (4) – heating in the temperature range 293 ÷ 493 K in the free state.

It should be noted that the repeated cycle leads to a similar "picture", which cannot be said for other methods of processing semi-finished products, where during the thermal cycling through the intervals of martensitic transitions in the loaded state noticeable irreversible deformations accumulate. As a rule, irreversible deformations accompanying martensitic transitions are undesirable and, as a result, worsen the dimensional stability of SME materials. It has been experimentally established here that the production of SME upon heating after cooling in the direct MT interval under pinched conditions is observed with zero deformation effect in the final heating interval.

Table 1 below shows the deformation responses as the average of the measurements for three samples at different levels of loading intensity.

**Table 1.** Deformation responses at various levels of loading intensity.

| Deformation response | Loading Intensity $\tau$, MPa |
|----------------------|-------------------------------|
| $\gamma_A$, %        | 100  200  250  300  350  400 |
| 1.44±0.06            | 3.52±0.08  4.60±0.09  6.11±0.13  10.20±0.15  11.65±0.15 |
| $\gamma_m$, %        | 0.43±0.03  0.65±0.05  0.58±0.06  1.51±0.08  1.60±0.08  3.31±0.10 |
| 1.01±0.03            | 2.87±0.05  4.02±0.07  4.60±0.07  7.60±0.10  8.34±0.11 |

3. Conclusion
As mentioned above, the process of setting the desired shape for the semi-finished product is an important technological stage in the production of devices from alloys with the shape memory effect. The application of the proposed method is possible in metallurgy, in particular to products from alloys
with a shape memory effect, and can be used in power engineering, construction, instrument making, and medicine.

References

[1] Tihonov A C, Gerasimov A P and Prohorova I P 1981 Primemnenie Effecta Pamjati Formi v Sovremennom Mashinostroenii [Application of the Shape Memory Effect in Modern Engineering] (Moscow: Mashinostroenie) p 80

[2] Andronov I N, Bogdanov N P, Vlasov V P and Severova N A 1998 Bulletin of Tambov State University Vol 3 3 236–238

[3] Andronov I N, Verbakhovskaya R A, Ovchinnikov S K and Severova N A 2007 Effects of reversible shape memory and thermocyclic return of deformation in TN-1 alloy Zavodskaya Laboratoriya [Industrial Laboratory] vol 73 2 64–67

[4] Patent 2310696 2007 MPKC22F 1/18, publ. 20.11.2007. Bul. 32

[5] Andronov I N, Churilina I V, Kryuchkov S V and Bogdanov N P 2019 The effect of preliminary thermal cycling under load on the magnitude of the deformations driven by the shape memory effect Zavodskaya Laboratoriya. Diagnostics of materials vol 85(7) 55–63 [In Russ.]

[6] Kryuchkov S V Bogdanov N P, Malinin V G, Savich V L and Konovalov M N 2019 IOP Conf. Ser.: Mater. Sci. Eng. 656 012029

[7] Vlasov V P, Andronov I N, Kakulia Yu B 1996 Utility Model Certificate No. 1538 of the Russian Federation, G01N 3/08. Installation for testing samples of materials with a complex stress state Application 94007969/28, 05. 03. 1994, publ. 01/16/1996

[8] Likhachev V A, Kuz’min S A and Kamenskaya Z P 1987 Effect Pamyati Formy [Shape Memory Effect] (Leningrad: LGU) p 216

[9] Likhachev V A and Malinin V G 1993 Strukturno-analiticheskaya Teoriya Prochnosti [Structural Analytic Theory of Strength] (Saint-Petersburg: Nauka) p 471