Production and plugging of TiNi SMA tube shell on a screw rolling mill

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In the present article the process of a tube shell producing from TiNi shape memory alloy rods and subsequent plugging are described. The deformation-temperature conditions of a shell plugging from an outer diameter of 38 to 26 mm and a tube schedule form 10 mm to 3.3 mm on a floating mandrel is considered in detail. The design features of the rolling mill and the tools used are analyzed. TiNi SMA seamless tubes with various outer diameters and pipes schedule were produced. During the production cycle mechanical and functional properties of SMA pipes are stable and are comparable with properties of initial SMA rods. Suggested technological scheme of TiNi seamless tubes productionn is promising in terms of the low metal consumption, simplicity of applied technological processes and tubes price reduction.

Key words: shape memory alloy, TiNi, tube, plugging, screw rolling, shell

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Introduction

Titanium nickelide TiNi-based shape memory alloys (SMA) semi-finished products have found wide practical application as blanks for the manufacture of various smart devices operating based on the shape memory effect [1–6]. One of the actively applied TiNi SMA semi-finished products is a seamless tube, used to produce smart sealing couplings for the aerospace and automotive industries, as well as vascular stands for medicine [7–11].

Tubes made of TiNi-based alloys have great potential for use as actuators and connecting elements, as they are simple and reliable in construction. These elements can generate high stresses and provide excellent mechanical performance [12]. Commonly, tubes that are used for engineering applications have a diameter of 20 mm or more. The demand for shape memory alloy with high properties [13–15] and complex geometry, including tubes, is constantly growing and requires the development of new manufacturing approaches [15–16].

The production of TiNi seamless tubes is a complex technological task due to the limited technological plasticity of titanium nickelide and the lack of developed deformation modes and heat treatment operations of shells production.

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Most of the world’s manufacturers are focused on the production of TiNi seamless tubes for medicine. The technological scheme for obtaining medium and small diameter tubes includes a deep drilling operation at the first stage, subsequent cold drawing with intermediate annealing in a protective atmosphere, as well as various finishing operations [16–21]. Detailed scientific and technological aspects of tube production remain uncovered as they are the Know-How of corporations.

Described technological scheme is suitable for obtaining seamless tubes for the subsequent producing of medical devices, in particular to produce cardiovascular stents [21]. However, the application of this technology for technical products is limited due to its high cost and low metal utilization rate. In addition, this technology does not provide the possibility to produce tubes with a diameter of more than 20 mm and a tubes schedule of more than 1 mm. The production of large-diameter tubes by drawing is limited by the required deformation force and very high load on the equipment. Consequently, the development of an alternative technological scheme for obtaining TiNi SMA seamless tubes is at a great interest.

Seamless tubes of various metals and alloys are commonly produced by screw rolling of a prefabricated shell on screw rolling mills [18]. Shells, in their turn, are commonly produced by the operation of piercing on two- or three-roll mils. The initial billet for shells production is a rod, which is usually obtained from a cast blank by the screw rolling method [21]. The operation of piercing of TiNi rods is limited due to the mechanical behavior of TiNi alloys.

Therefore, in the present article, alternative technological scheme for the obtaining of a TiNi SMA seamless tubes with minimization of metal losses and higher efficiency is performed. Studies of the structure, mechanical and functional properties of the obtained seamless tubes at various stages were also conducted. From the results obtained several major fundings may be highlighted. The deformation-temperature conditions of a shell plugging from an outer diameter of 38 to 26 mm and a tubes schedule form 10 mm to 3.3 mm on a floating mandrel is considered in detail. The design features of the rolling mill and the tools used are analyzed. With the application of developed technological scheme TiNi SMA seamless tubes with various outer diameters and tubes schedule were produced. Properties od obtained TiNi tubes are stable on all production stages.

Materials and methods

Ti – 50.0 at.% Ni alloys hot-rolled rod with a diameter of 40 mm was used as the initial blank. SC 703 electrical discharge super drill with a copper electrode of 2.4 mm in diameter and 400 mm in length and a high-precision SCT32-ST electrical discharge machine operating with a molybdenum wire of 0.16 mm in diameter were used for the obtaining of shells from the initial blank. Screw rolling was carried out on a SVP 70 screw rolling mill. Samples for structural-phase analysis as well as for the determination of mechanical and functional properties at various stages of rolling were cut out from the resulting tubes by the electrical discharge method. Annealing at the 750 °C for 30 min with water colling was served as a reference treatment (RT). Samples for light microscopy were ground on abrasive paper with a grain size from P120 to P2500 with subsequent polishing. These samples after mechanical grinding and polishing, as well as samples before conducting of *X*-ray analysis, were etched in a solution of $1HF : 3HNO_3 : 6H_2O_2$.

The microstructure and phase composition of the alloys were assessed by optical microscopy using a Versamet-2 Union optical microscope with a magnification of 50 to 100 and *X*-ray diffraction analysis using a DRON-3 *X*-ray diffractometer in Cu K_α radiation in a 2θ angel range from 37 to 47°. Mechanical properties were studied by hardness and tensile tests. Hardness measuring was done on a LECOM 400-A hardness tester under a load of 1 N with at least 10 indentations for each sample and determination of the average hardness value. Tensile tests were performed using an INSTRON 2253 universal testing machine at a strain rate of 2 mm/min. Flat samples with dimensions of 1×2×50 mm were applied for tensile tests and three samples for each condition were tested. The functional properties were studied using differential scanning calorimetry using a Mettler Toledo DSC 3+ calorimeter with a heating and cooling rate of 10 °C/min in the temperature range of –100–100 °C to determine the characteristic temperatures of martensitic transformations (MT) and a special thermomechanical method for determining completely recoverable strain after deformation by bending, which consisted in inducing deformation below the temperature of the starting of the forward MT M_s, and subsequent heating above the temperature of the finishing of the reverse MT A_f to implement the shape memory effect and fix the value of recoverable and residual strains. Deformation was induced at the room temperature.

Obtaining of TiNi SMA shell

At the first stage of the development of a new approach to the manufacturing of TiNi seamless tubes the method for obtaining of shells from initial rods with the lower metal consumption as compared to the deep drilling process is determined. After the analysis of possible technological solutions electrical discharge cutting (EDC) is chosen as the most appropriate method.

The applied technological scheme for shell obtaining by EDC includes drawing of a working sketch in order to minimize metal consumption **(Fig. 1)**, cutting out the through hole with a diameter of 3 mm in the initial blank with a length of 250 mm on a SC 703 electrical discharge super drill and subsequent cutting for two contours on a SCT32-ST electrical discharge machine in order to obtain a shell and a TiNi rod with the diameter, slightly lower than the inner diameter of obtained shell. These technological operations allow reducing possible defects and maintaining the coaxiality and profile of the resulting shell with the noticeable lower metal consumption as compared to deep drilling. With the application of described scheme, a TiNi SMA shell with an outer diameter of 40.0 mm and a tubes schedule of 10.0 mm **(Fig. 2)** was obtained for subsequent plugging and a rod with a diameter of 14.5 mm, which can be used for subsequent processing, for ex. forging or drawing to obtain a rod or a wire, as well as used as a blank to produce TiNi SMA seamless tubes of a smaller diameter by the application of developed technological scheme.

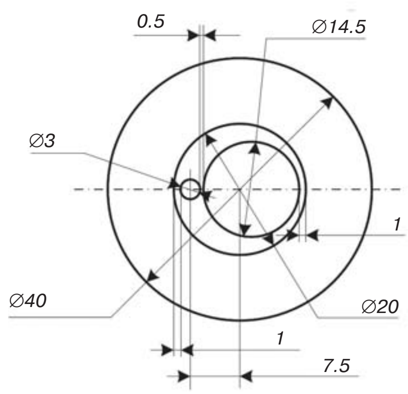

Fig. 1. Working sketch for a shell cutting

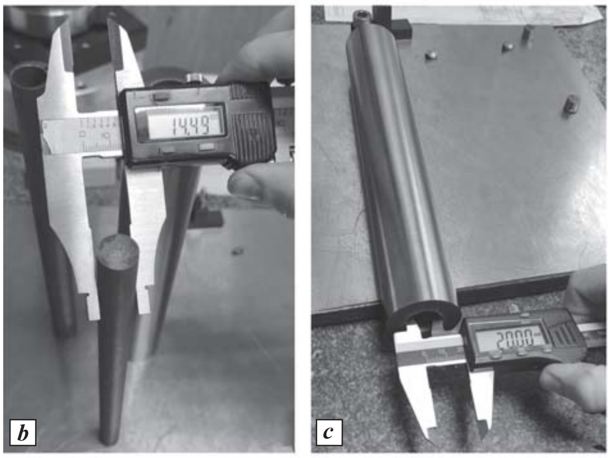

Fig. 2. General views of the upper edge of a TiNi SMA shell (*a*), TiNi SMA shell with an outer diameter of 40.0 mm and a tubes schedule of 10.0 mm (*b*), TiNi SMA rod with a diameter of 14.5 mm (*c*) after cutting on a SCT32-ST electrical discharge machine (*photo by authors*)

Experimental plugging of a TiNi SMA shell on a screw rolling mill

Plugging of obtained TiNi SMA shell was carried out on a SVP 70 screw rolling mill at the deformation temperature of 900 °C, that are the most commonly applied for the hot deformation of TiNi SMA [22—25]. A stainless steel rod of grade 4X5MFS with a diameter of 18.5 mm was used as a floating mandrel. Laundry soap was used as a lubricant for the mandrel.

The rolling route of the TiNi SMA shell and the dimensions before and after each pass are presented in **Table 1**. The general appearance of the workpiece during the rolling process and after the first pass is shown in **Fig. 3**.

After each pass, 80—90 mm long samples are cut from the shell to study the formation of the structure and properties during the rolling process. In order to study the possibility of performing the sizing reduction operation the last pass is carried out on the mandrel only partially. As a result, a seamless tube with various diameters and tubes schedule is obtained.

It should also be noted that after the last pass, a "triangle"-shaped defect appeared at the trailing end of the tube that was rolled on the mandrel (plugging process), consisting of a compression on three sides of the tube without the formation of through cracks. This defect indicates the maximum-thin wall thickness for rolling on the SVP 70 rolling mill while maintaining the technological plasticity of titanium nickelide **(Fig. 4)**.

After plugging and sizing reduction operations, samples were cut out from the resulting seamless tubes for the study of the structure and properties formation at various stages of screw rolling. The cross-

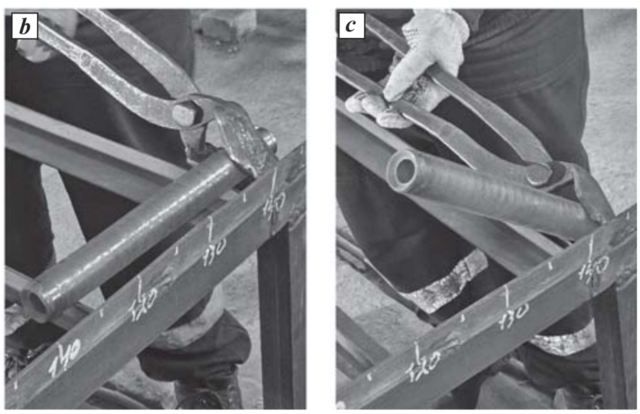

Fig. 3. General views of the screw rolling mill SVP 70 (*a*), the TiNi SMA shell during plugging on the SVP 70 screw rolling mill (*b*) and after the first pass (*c*) (*photo by authors*)

Table 1
Rolling route of a TiNi SMA shell

Pass No	Heating time, min	*D*/*H*/*L** before rolling,mm	Relative strain ε, %	*d*/*h*/*l** after rolling, mm	Designation
1	35	40.0/10.0/250	19	35.7/8.8/340	1
2	15	35.7/8.8/260	15	33.0/6.4/325	2
3	10	33.0/6.4/330	25	28.0/5,3/400	3
4	5	28.5/5.3/300	21	25.5/3,3/258	4–1
			–	24.0/5.0/140 (sizing reduction)	4–2

**D*/*H*/*L* and *d*/*h*/*l* – Outer diameter/ tubes schedule/length.

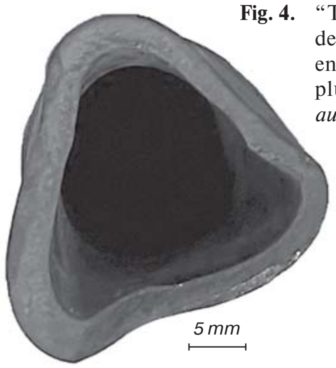

Fig. 4. "Triangle"-shaped defect at the trailing end of the tubes after plugging (*photo by authors*)

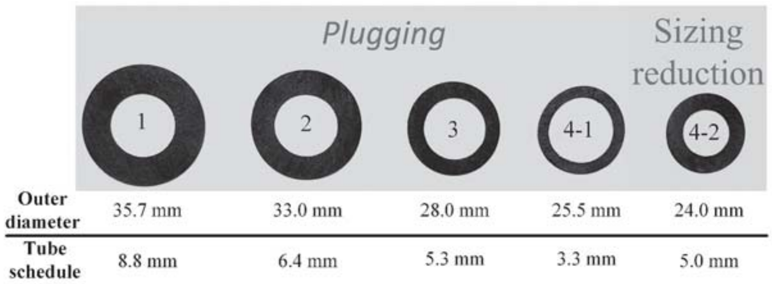

Fig. 5. Cross section of obtained TiNi SMA seamless tubes

section of obtained TiNi SMA seamless tubes is shown in **Fig. 5**.

Structural-phase state TiNi SMA seamless tubes

The results of the *X*-ray analysis of the TiNi SMA seamless tubes are presented in **Fig. 6**.

Based on the results obtained the positions of the main *X*-ray lines peaks were determined, as well as the full width at a half height (FWHM). The results of these measurements are presented in the **Tables 2, 3** in comparison with the initial state of the alloy after RT. The error limits of obtained values is ±0.05.

The analysis of the results obtained shows that rolling of the TiNi SMA seamless tubes does not lead to a noticeable change in the phase composition of the alloy at room temperature. Regardless of the pass number, the main phase is B19′ -martensite. The presence of a small amount of R-phase and B2-austenite is also indicated. A noticeable shift in the position of the martensite line peaks is observed only after the 4rd pass (4–1) for the line $(110)_{B19'}$, as well as and the maximum broadening of the *X*-ray line $(020)_{B19'}$. A higher strain hardening after this screw rolling pass may be associated with a thinner schedule of the resulting pipe leading to the faster cooling as compared to other obtained tubes.

The images of the structure of TiNi SMA seamless pipes depending on the screw rolling pass number, obtained by optical microscopy, are shown in **Fig. 7**.

Analysis of the obtained structure images does not allow to define its qualitative changes depending on the screw rolling pass number. In addition, due to the fact that at room temperature the main phase in all studied samples is martensite, grain boundaries cannot be identified, and the conclusion about the change in the size of the structure elements cannot be provided.

DSC curves of studied TiNi SMA seamless tubes depending on the screw rolling pass number are shown in **Fig. 8**. The defined characteristic temperatures of martensitic transformations are presented in **Table 4**.

Analysis of the obtained results allows us to conclude that rolling of the TiNi SMA seamless tubes does not lead to a sharp shift in the temperatures of the forward and reverse martensitic transformations (MT), as well as in the type of calorimetric curves as compared to different deformation modes. Forward and reverse transformations

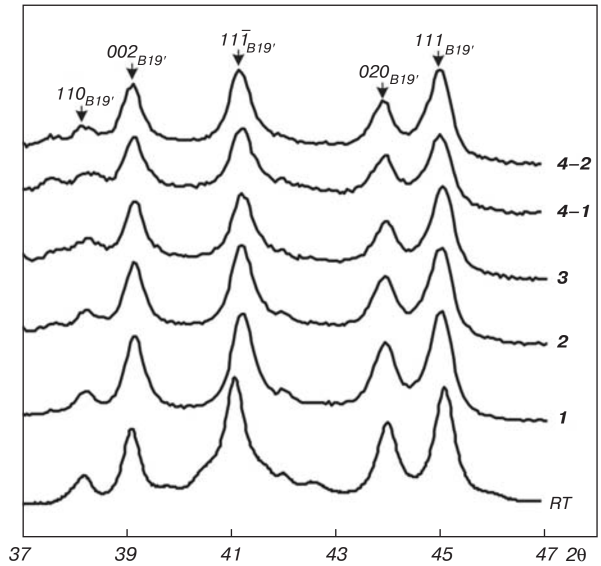

Fig. 6. *X*-ray diffraction patterns of TiNi SMA seamless tubes depending on the screw rolling pass number

Table 2
Position of *X*-ray line peaks depending on the screw rolling pass number

Sample No	*X*-ray line peaks				
	110	002	$11\bar{1}$	020	111
0 (RT)	38.17	39.08	41.04	43.97	45.04
1(TN-1 35.1 mm)	38.19	39.10	41.22	43.90	45.01
2(TN-1 32.4 mm)	38.24	39.10	41.17	43.90	45.01
3(TN-1 27.8 mm)	38.19	39.10	41.17	43.95	45.01
4-1(TN-1 26.0 mm)	38.34	39.10	41.17	43.90	44.97
4-2(TN-1 23.6 mm)	38.09	39.10	41.12	43.85	44.97

Table 3
FWHM of *X*-ray line peaks depending on the screw rolling pass number

Sample No	110	002	$11\bar{1}$	020	111
0 (RT)	0. 56	0.43	0.56	0.54	0.51
1(TN-1 35.1 mm)	0.68	0.54	0.61	0.73	0.61
2(TN-1 32.4 mm)	0.80	0.61	0.70	0.76	0.67
3(TN-1 27.8 mm)	0.70	0.76	0.79	0.75	0.66
4-1(TN-1 26.0 mm)	0.60	0.98	0.96	1.02	0.78
4-2(TN-1 23.6 mm)	0.60	0.70	0.75	0.88	0.66

Table 4
Characteristic tempezratures of martensitic transformations of TiNi SMA seamless tubes depending on the screw rolling pass number

Sample No	M_s, °C	M_p, °C	M_f, °C	A_s, °C	A_p, °C	A_f, °C	A_s–M_f, °C
0 (RT)	68	60	48	75	92	98	50
1	66	51	36	74	83	98	62
2	66	54	40	74	86	97	57
3	63	52	40	75	85	96	56
4-1	61	51	41	74	85	94	53
4-2	63	52	40	73	85	93	53

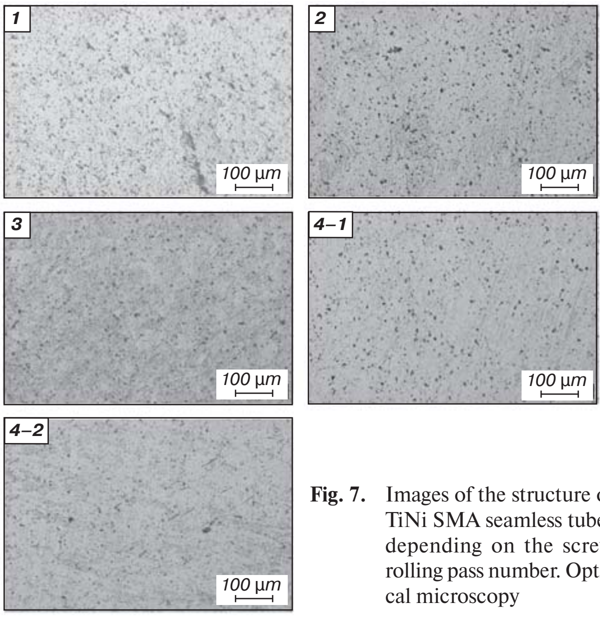

Fig. 7. Images of the structure of TiNi SMA seamless tubes depending on the screw rolling pass number. Optical microscopy

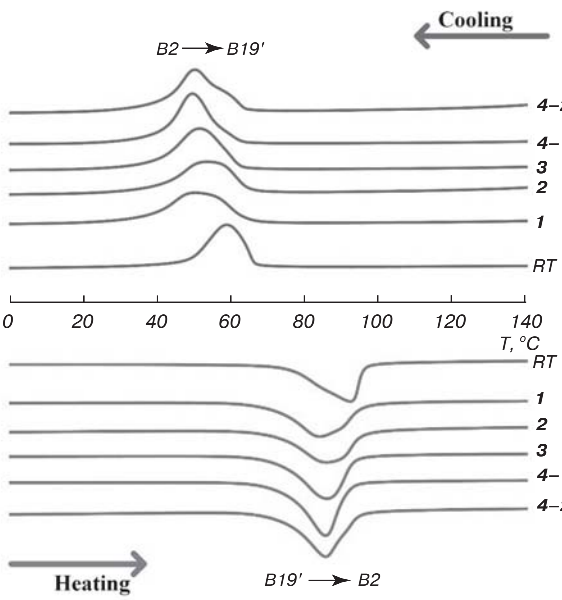

Fig. 8. Calorimetric curves of TiNi SMA seamless tubes depending on the screw rolling pass number

occur in all cases in one stage in a relatively narrow temperature range. When comparing the temperatures of forward and reverse MT, a noticeable decrease of 8-10 °C is observed in the region of lower temperatures as compared to RT. Both during heating and cooling, an asymmetry in the calorimetric peak is seen, indicating the occurrence of the transformation through the intermediate R-phase. This indicates an increase in the number of defects in the crystal structure due to deformation.

Mechanical properties of TiNi SMA seamless tubes

The hardness tests results of TiNi SMA seamless tubes depending on the screw rolling pass number and a zone of a tubes cross section are presented in **Table 5**. The mechanical characteristics obtain by tensile tests are presented in **Table 6**. Typical tensile curves and the average hardness values are shown in **Fig. 9**.

Based on the results obtained it may be concluded that significant changes in the value of ultimate tensile strength, dislocation yield stress and hardness is not determined after various stages of screw rolling. Slight increase of the mechanical characteristics after the last two screw rolling passes can be attributed to the accumulated strain hardening while the decrease in the hardness value after first two crew rolling passes as compared to RT is explained more pronounced deformation heating. Thus, the analysis of structural-phase state and mechanical properties of equiatomic TiNi SMA seamless tubes showed that hot screw rolling is not accompanied by a significant change in hardness, structural state and characteristic temperatures of martensitic transformations, which indicates the correct choice of the rolling process route in

Table 5
Results of hardness tests of TiNi SMA seamless tubes depending on the screw rolling pass number and a zone of a tubes cross section

Cross section area	Sample No				
	1	2	3	4-1	4-2
Outer diameter	196	191	224	213	208
Half edge of a tube schedule	221	200	230	222	220
Inner edge of a tube schedule	210	191	224	210	227

Table 6
Mechanical properties of TiNi SMA seamless pipes depending on the screw rolling pass number and a zone of a tubes cross section

Treatment	σ_{cr} (MPa)	σ_y (MPa)	$\Delta\sigma$ (MPa)	$\sigma_в$ (MPa)	δ (%)	HV
0(RT)	200	535	335	844	29	230
1	193	565	372	835	48	210
2	199	560	361	867	55	194
3	185	615	430	889	58	226
4-1	192	610	413	922	56	216
4-2	215	630	415	954	52	219

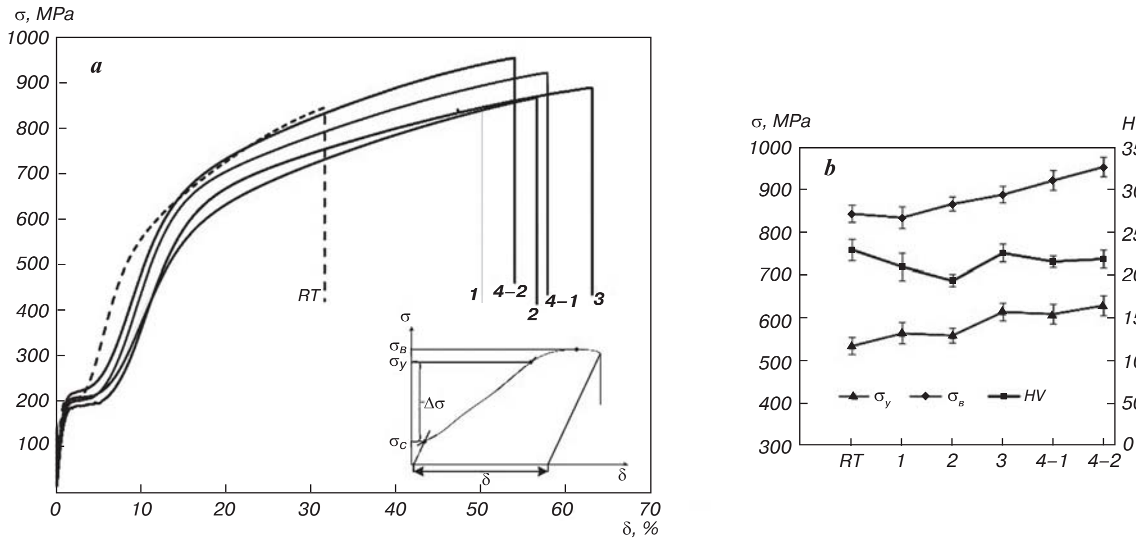

Fig. 9. Typical tensile curves (*a*) and the average values of hardness, dislocation yield stress and ultimate tensile strength (*b*) of TiNi SMA seamless tubes depending on the screw rolling pass number

terms of preserving the properties of the alloy and the possibility of subsequent additional processing. The obtained results reveal that developed technological scheme for the obtaining of the shell and subsequent production of TiNi SMA seamless tubes by screw rolling on a floating mandrel is promising in case of fabrication of tubes with stable structure and properties in combination with the low metal consumption, simplicity of applied technological processes and tubes price reduction

Conclusions

In the present study the technological scheme for the production of TiNi SMA seamless tubes are developed. The process of a shell producing from TiNi shape memory alloy rods and subsequent plugging are described. During the production cycle mechanical and functional properties of SMA tubes are investigation. Based on the results obtained main conclusions are:

1. TiNi SMA seamless pipes with an outer diameter from 38 to 26 mm and a pipe schedule form 10 mm to 3.3 mm on a floating mandrel is produced by application of electrical discharge method for the shell obtaining and subsequent hot screw rolling at a temperature of 900 °C for plugging and sizing reducing operations.

2. Hot screw rolling of TiNi SMA seamless tubes to the tube schedule of 3.3 mm on the SVP 70 rolling mill leads to the appearance of "triangle"-shaped defect at the trailing end of the tube indicating the maximum-thin wall thickness for rolling on this equipment while maintaining the technological plasticity of titanium nickelide

3. The analysis of structural-phase state and mechanical properties of equiatomic TiNi SMA seamless tubes showed that screw rolling is not accompanied by a significant change in hardness, structural state and characteristic temperatures of martensitic transformations, which indicates the correct choice of the rolling process route in terms of preserving the properties of the alloy and the possibility of subsequent additional processing.

4. Suggested technological scheme of TiNi seamless tubes production is promising in terms of the low metal consumption, simplicity of applied technological processes and tubes price reduction.

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